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An evaluation of the physical and chemical properties of high-calcium fly ashes in Iowa

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An evaluation of the physical and chemical properties of high-calcium fly ashes in Iowa

by

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INTRODUCTION

Fly ash is a finely divided particulate material that is removed from the flue gases of pulverized coal burning power plants. Recent emphasis on the utilization of coal to meet our growing energy demand has created a serious waste disposal problem.

On a worldwide basis, the United States ranks second in annual fly ash production (see Figure 1). Only the Soviet Union produces more fly ash than the United States. Current estimates place worldwide fly ash production at about 3.2×10^8 U.S. short tons (2.9×10^8 metric tons) per year [95].

The United States produces approximately 50 million tons of fly ash each year [2, 30]. About 20% of the fly ash is currently being utilized by various industries so roughly 40 million tons of ash remains to be disposed of each year [2]. Coal combustion by-products rank high on the list of bulk solid wastes that are produced annually in the United States. Several researchers have expressed concern about the toxicity of trace elements that are commonly found in fly ash [2, 24, 67, 98]. Production and utilization trends for U.S. fly ash are shown in Figure 2.

Utilization of fly ash has been dismal at best. The exact reason for the poor utilization of fly ash is unclear; perhaps the stigma of being called a "by-product" has limited its usage. The construction industry is the only major industry that currently utilizes large amounts of fly ash. The major use of fly ash in the U.S. is as a cement replacement in portland cement concrete products. The construction industry also routinely utilizes fly ash as a soil stabilization/amendment agent, a grouting admixture, as a mineral filler in asphalt pavement construction and for several other minor miscellaneous uses [1, 17, 30, 95]. Many researchers have shown interest in extracting selected elements from fly ash (generally Al, Ti and Fe are of main interest) [9, 13, 99]; however, few extraction plants have been built and some of those that were built have been economic failures [15]. However, it is obvious that the economic appeal of such extraction processes will increase significantly in future years because of decreasing availability of natural resources.

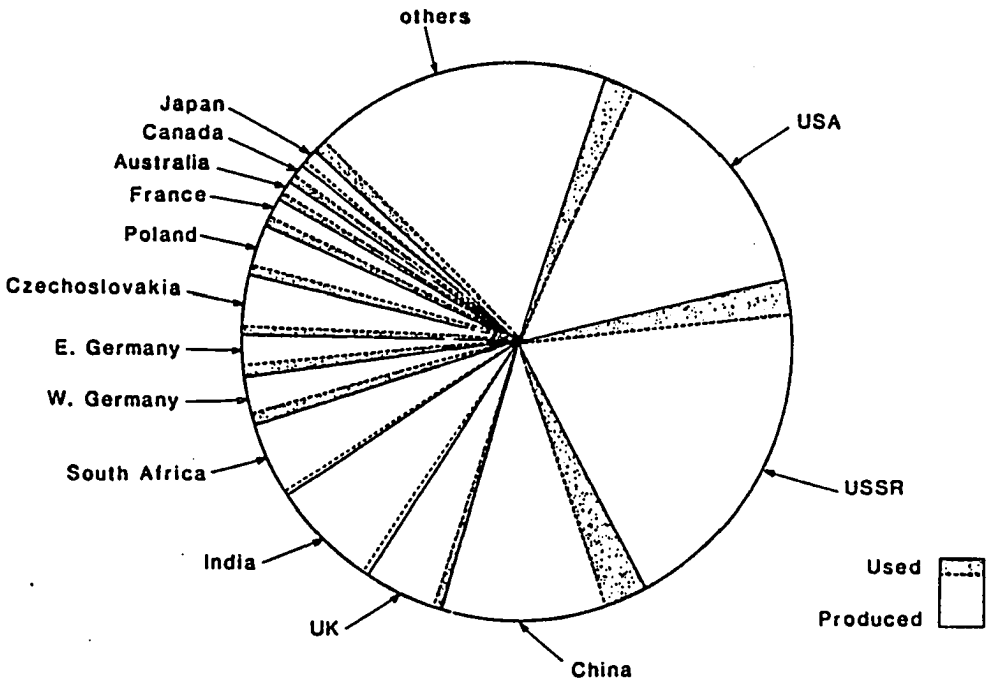


Figure 1. Recent worldwide annual production of fly ash and its utilization as an admixture in cement and concrete (Dihr, as shown in Swamy [95])

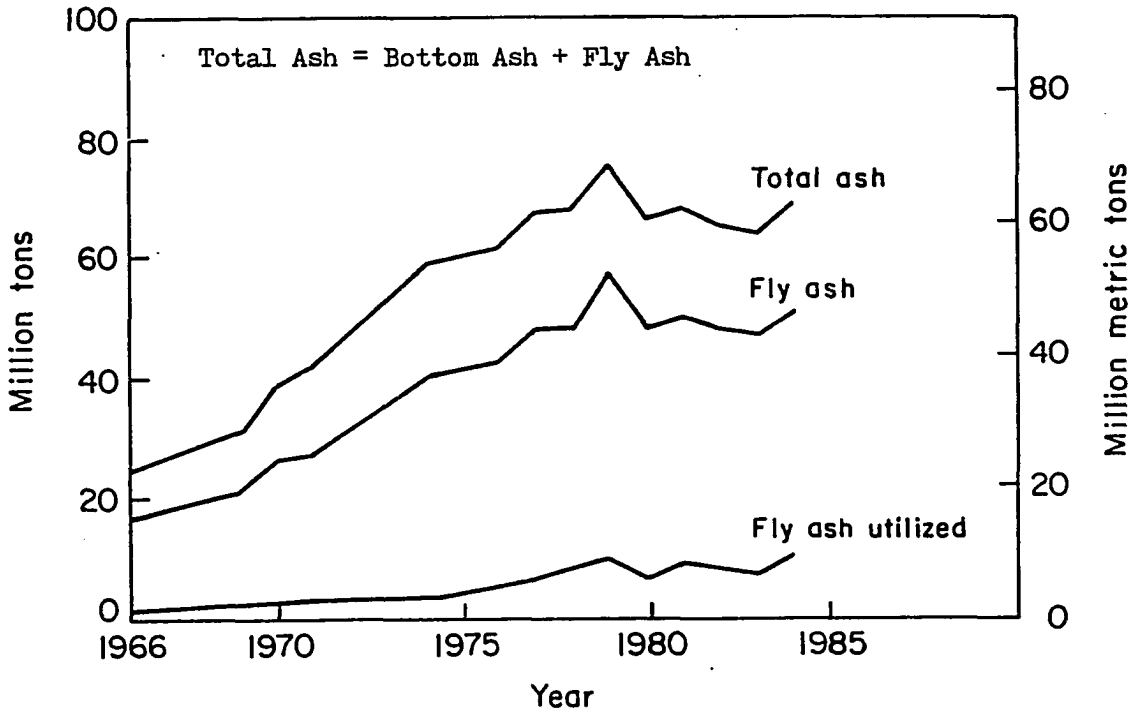


Figure 2. *Quantity of fly ash produced and utilized in the United States [30]*

Governmental efforts have helped to increase fly ash usage. Since 1982, when the Environmental Protection Agency (EPA) produced guidelines for the utilization of fly ash in federally funded construction projects, usage has increased [30]. However, since the mandate only applies to cement and concrete products, there is little hope of increasing fly ash utilization above 50% because the United States only produces about 85 million tons of portland cement each year [62]. Hence, without the development of new uses for fly ash, much of it is still destined for disposal.

The Iowa Fly Ash Situation

Iowa is heavily dependent on electricity generated from the burning of coal. Initially, many municipalities built small power plants to meet the demands of their immediate areas. Such power plants were rather small and produced only small amounts of fly ash. However, since the oil embargo of 1974, electrical energy production has become a big business. Most of Iowa's large electrical generating facilities were built during the late 1970s and early 1980s. A breakdown of the total generating capacity of the State of Iowa is shown in Figure 3. It is evident that these large power plants, all of which came on line between 1977 and 1983, clearly dominate electrical power production in the state. Since these power plants produce the most electricity they also produce the most fly ash. The rate at which a modern, relatively large power plant produces fly ash is nearly overwhelming; a 675 MW power plant will consume about 435 tons of pulverized coal in one hour, and in doing so, will produce about 23 tons of fly ash. The fly ash production and utilization trends for Iowa are shown in Figure 4. These statistics have been compiled by the Materials and Environmental Research Laboratory (MERL) at Iowa State University, on a yearly basis since 1983, as part of the research presented in this dissertation. Fly ash production and utilization prior to 1983 was uncertain.

Utilization of Iowa fly ashes has increased significantly over the past five years. Iowa currently utilizes about 40 to 50% of its fly ash each year. However, existing markets for fly ash

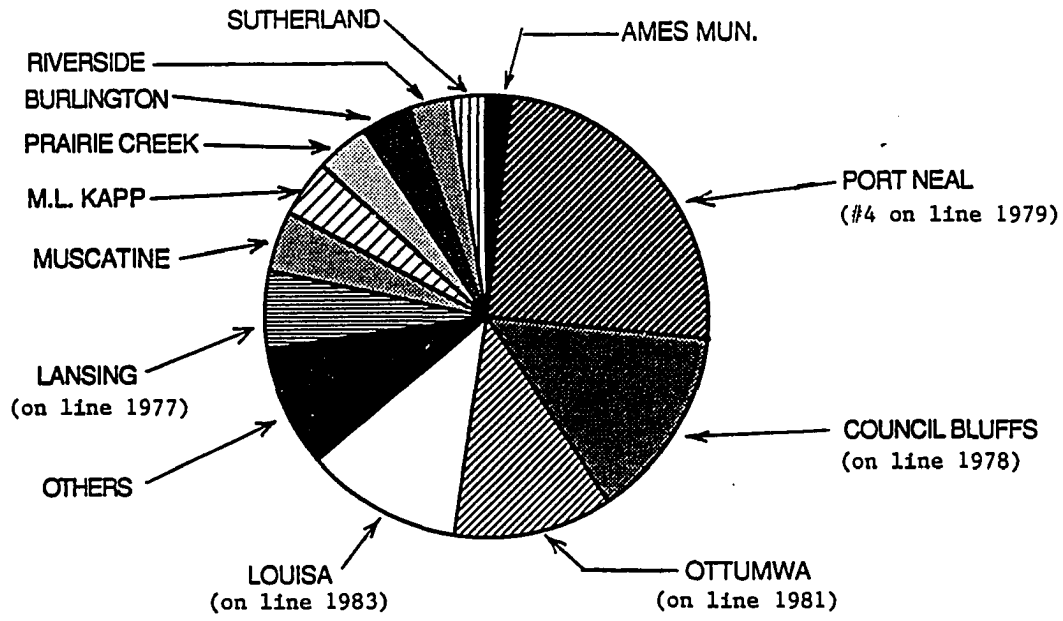


Figure 3. A breakdown of the generating capacity of coal burning power plants in Iowa

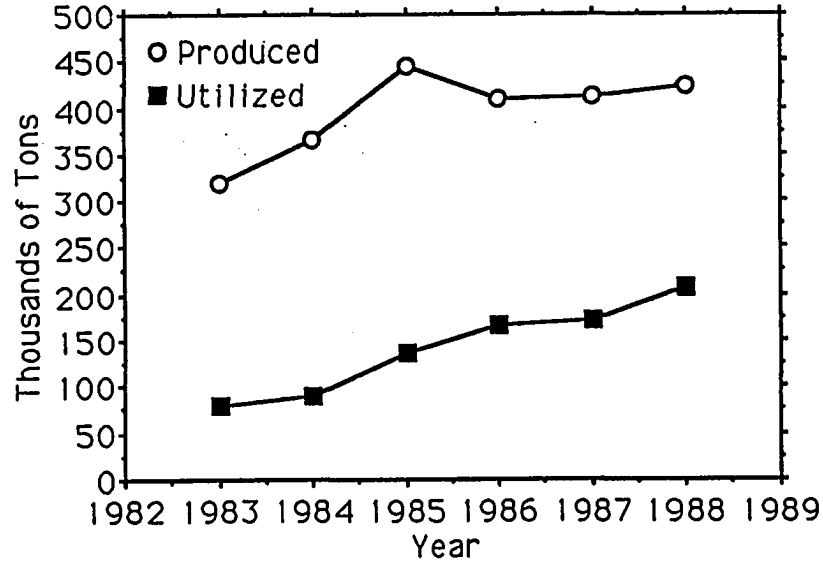


Figure 4. Production and utilization trends for Iowa high-calcium fly ashes

utilization are nearly saturated so little if any gain in utilization can be expected in the near future unless: (1) larger replacements of fly ash for portland cement are allowed in concrete construction; (2) new innovative methods of utilization are invented; or (3) inexpensive methods of stock piling fly ash are researched and developed.

The Iowa Fly Ash Affiliate group was established in 1984 to address the Iowa fly ash utilization/disposal situation. Much of the growth in fly ash utilization (see Figure 4) can be directly attributed to the research sponsored by the Iowa Fly Ash Affiliates. Currently the Affiliate program is actively involved in the basic studies needed to convince state and local authorities that fly ash is a resource that should be recycled rather than a by-product that must be disposed of.

Scope and Objectives

The scope of this study is limited to fly ashes that are currently being produced in large volumes in the state of Iowa. Although this appears to be a very narrow topic, in reality the study should be applicable to fly ashes that are currently being produced in many other mid-western and far-western states.

The major objective of this research was to actively pursue the characterization of Iowa fly ashes in the hope that better knowledge of their physical and chemical properties would lead to increased utilization. Many tasks were defined during the duration of this research to help reach the major objective. The tasks can be enumerated as follows:

(1) To develop experimental methods (both physical and chemical) to accurately measure fly ash properties.

(2) To investigate relationships between the physical and chemical properties of fly ash.

(3) To compare existing fly ash testing methods that are currently endorsed by the American Society for Testing and Materials (ASTM) to those developed above.

(4) To study the influence of power plant operating conditions on the physical and chemical properties of fly ash.

All of the tasks defined above were conducted in a manner such that the results could be used to construct a database of fly ash properties. This database of fly ash "quality" was deemed as essential to establishing the long-term trends in fly ash properties. Such information will be pertinent in future years to engineers who may need to establish exact reasons for early distress or poor service life of various structures containing (or composed of) fly ash.

LITERATURE SURVEY

The purpose of this literature review is only to inform the reader about the major aspects of fly ash. It would be extremely difficult, if not impossible, to summarize all events related to the utilization and/or characterization of fly ash because the literature abounds with papers dealing with fly ash. Hence, abiding by the proposed scope of this dissertation as was defined earlier, only a very general description of fly ash will be presented. The majority of the information that will be presented deals with what fly ash is, rather than how it can be utilized. This bias stems from the author's belief that one of the major failures of early (and even recent) fly ash research was a lack of vision, a simple lack of knowledge about the fundamental constituents that collectively are referred to as fly ash. Fly ash is not a homogeneous, well-defined product. Rather, it is a by-product from the burning of coal; one must remember that power plants were designed to produce electricity, not fly ash. Perhaps Helmuth [30], from the Portland Cement Association, has best stated the inadequacies of fly ash research:

> Despite the fact that fly ash has been used in concrete for more than 50 years and extensive literature on the subject exists, it is apparent that such use has been based largely on empirical relationships of limited validity. There have been persistent problems, therefore, concerning the variability of fly ash and the difficulty of characterizing it for predictable performance in concrete. These problems are further complicated by the fact that much of what has been learned concerning bituminous coal fly ashes does not apply to subbituminous coal and lignite fly ashes, sources of which are being rapidly developed in the western United States.

Hence, characterization is the key to both understanding fly ash and also to increasing its utilization. Characterization is used in a very broad sense in this instance. It is meant to include details concerning the entire process of fly ash production. Thus, it should include information about: (1) the source of coal; (2) the power plant operating parameters; and (3) the properties of fly ash exiting the process. Obviously, the utilization of fly ash will also be dependent on the specifications drafted by interested parties who wish to ensure potential customers that their product (i.e., fly ash) will perform as promised.

Energy From Coal

Coal is a relatively heterogeneous mixture of both organic and inorganic constituents. For the purpose of this treatise, we will adopt a rather simple definition of coal [26]:

> Coal — a general name for a firm, brittle, sedimentary, combustible rock derived from vegetable debris which has undergone a complex series of chemical and physical changes during the course of many millions of years.

Coal is commonly classified by type, rank and grade [30]. Coal type refers to the different types of residual plant material present in coal; coal rank refers to the degree of coalification or metamorphosis experienced by the coal; and coal grade refers to the inorganic constituents (mineral matter) present in the coal. The classification of coal by rank provides the most useful information for the exploitation of coal for the production of electrical energy. Hence, the American Society for Testing and Materials (ASTM) constructed specification D 388 which classified coal into four categories based on rank [6]. The major categories designated by the ASTM are anthracitic, bituminous, sub-bituminous and lignitic. Each major category is further subdivided into smaller groups of coal that exhibit similar physical and chemical characteristics. Coal rank also roughly corresponds to both geologic age of the coal and the ultimate heating value of the coal as shown in Figure 5.

The United States has an excellent supply of easily exploitable coal reserves. The location and rank of the major coal fields in the contiguous United States are illustrated in Figure 6. Current estimates place the recoverable coal reserves at about 260,300 million short tons [89]. However, the United States also has a huge demand for electrical energy — energy that is often obtained from burning coal. Coal consumption has increased dramatically in the past several decades (see Figure 7). Also, the mode in which the coal has been consumed has been changing. The current trend indicates an increase in the amount of coal used to generate electricity. All other major coal consumers have reduced their demand for coal; hence, in 1988, about 85% of the coal consumed in the United States was used to generate electricity.

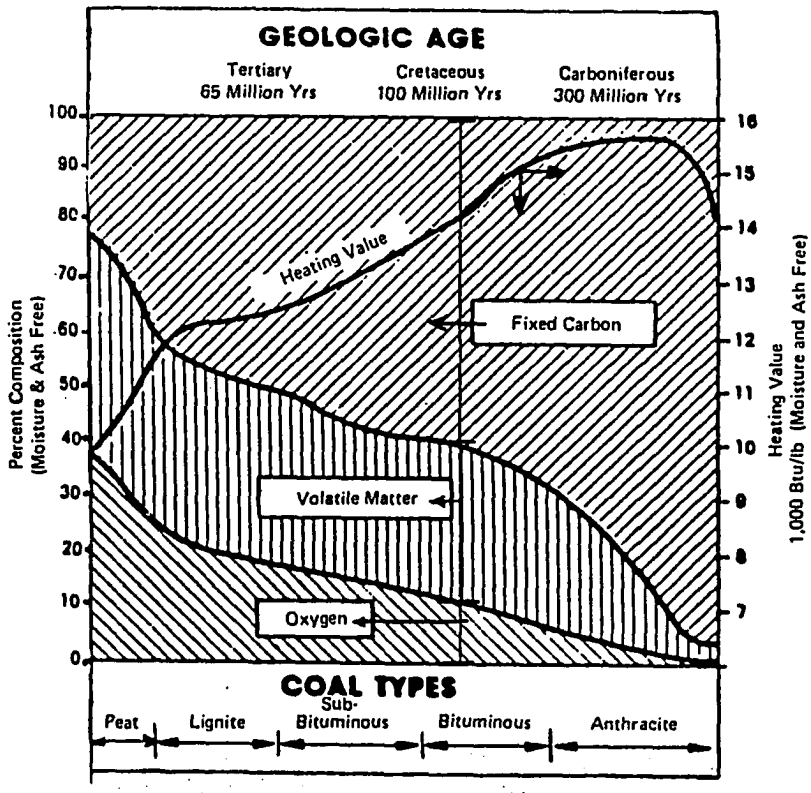


Figure 5. Geological age—coal type graph (DOE, 1979, as shown in Shannon [89])

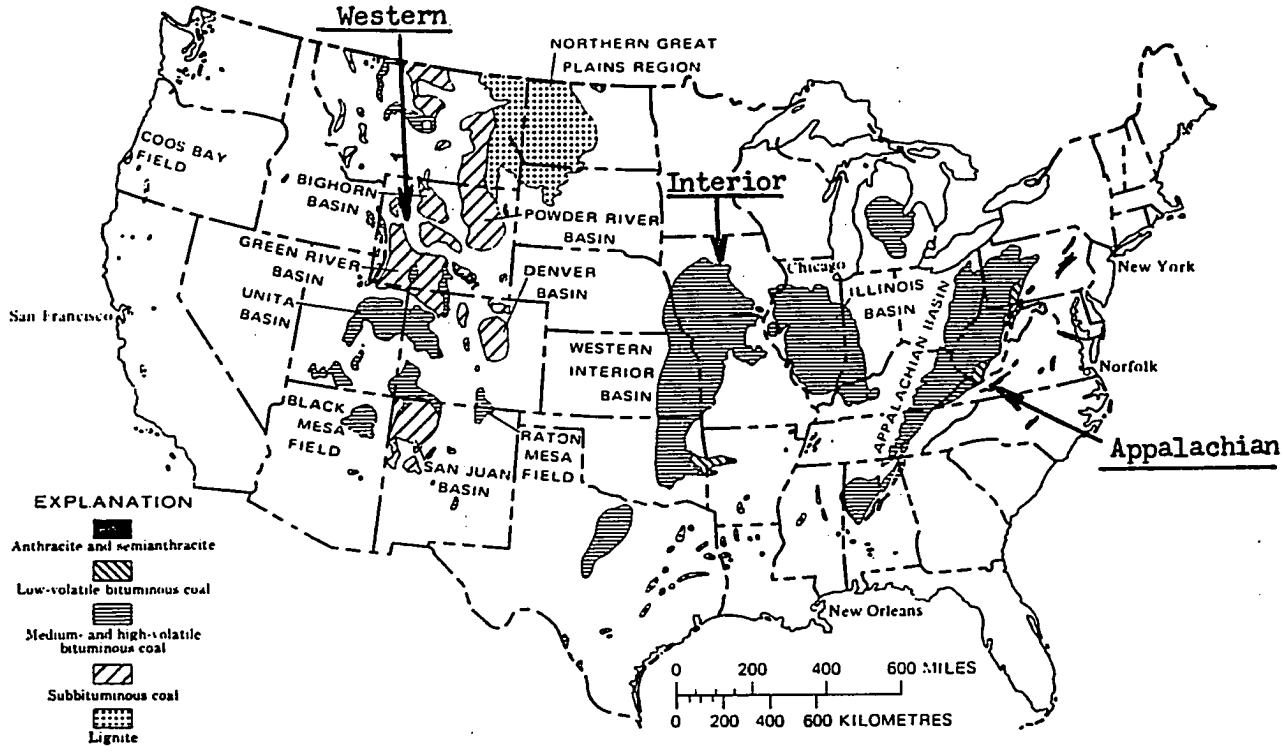


Figure 6. Coal fields of the continental United States (USGS Bull. 1412, as shown in [89])

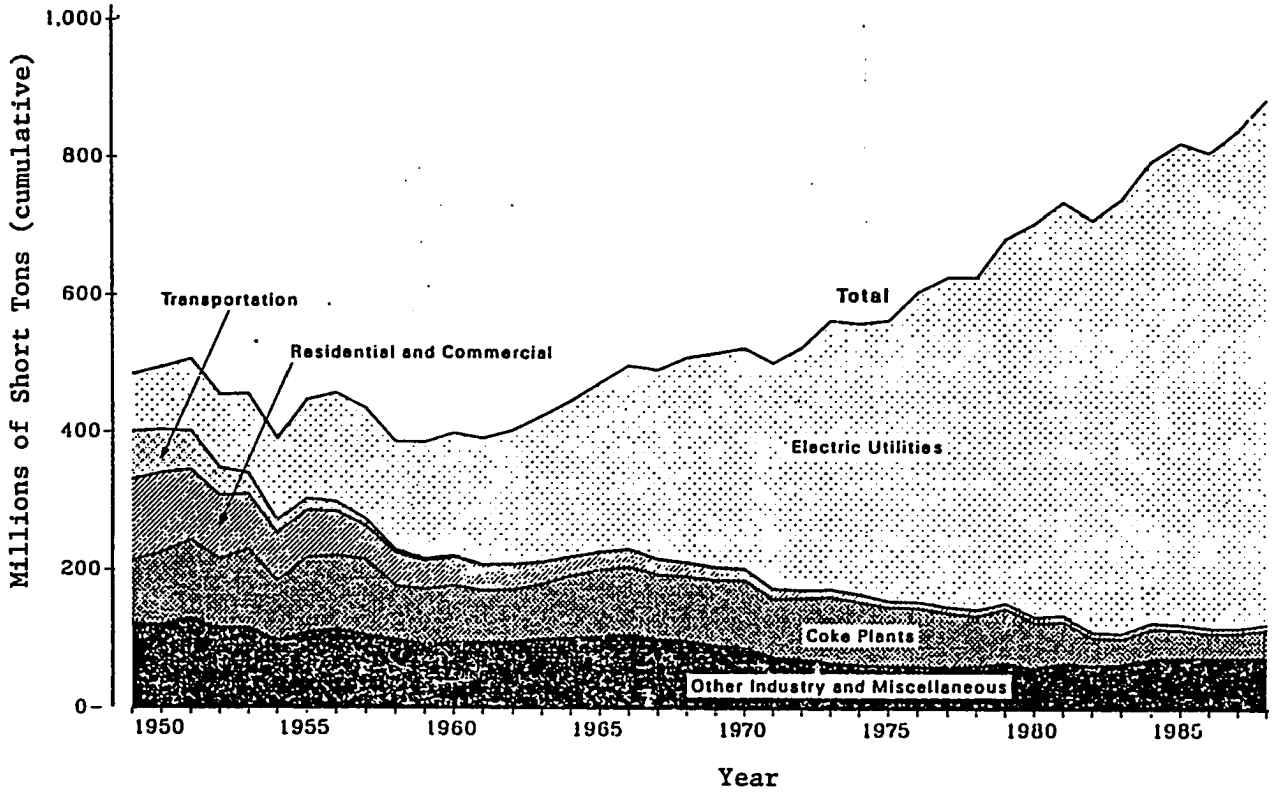


Figure 7. Coal consumption in the United States from 1949–1988 [7]

The location of the coal mines being exploited for energy production has also changed significantly in the last fifteen years. Prior to 1975, less than 15% of the coal consumed by the U.S. was obtained from mines west of the Mississippi river. By 1988, western coal production had more than doubled and it currently constitutes over 38% of the coal mined in the contiguous United States [7]. There are several reasons for this dramatic increase in the use of western coal for the generation of electrical power. First, coal has always been considered a rather "dirty" source of energy. Constant pressure from environmental groups concerned about the acid rain problem has caused a shift to the exploitation of coals that contain low concentrations of sulfur. Most western coal reserves are low in sulfur when compared to coals mined in the middle or eastern United States. Secondly, the coal mining industry has pursued different mining techniques in recent years. Most western coals can be effectively strip mined because they are not deeply buried in the earth. Strip mining requires the exploitation of vast areas of land; the eastern United States is simply too densely populated to allow strip mining on a scale comparable to that currently practiced in the sparsely populated western states. Third, economical modes of transportation were needed to move the huge amounts of coal to the power plants. The unit train concept coupled with the availability of railroad right-of-way in the western Unites States allowed for the rapid exploitation of these western coal reserves. A good example of this dramatic increase in demand for western coal can be illustrated by using information concerning coal mined in the state of Wyoming.

Wyoming contains ten major coal-bearing regions [41]. However, for the purpose of illustration, only two of the regions, the Powder River Basin and the Green River Basin, need to be considered because they account for about 95% of all the coal mined in the state [41]. Coal production in Wyoming increased from about 8 million tons in 1970 to over 140 million tons in 1985[41]. Currently, about half of all the coal mined west of the Mississippi River comes from Wyoming coal fields. The distribution of coal shipped from Wyoming to other states is shown in

Figure 8; it is obvious that there is a large demand for the low-sulfur, sub-bituminous coal that is mined in Wyoming.

The utilization of western coals for the production of electrical energy has also influenced the design aspects of pulverized coal burning power plants. Power plants that burn western coals need larger boilers than power plants of similar capacity that burn eastern (bituminous) coals, they also require adjustments in their particulate collection systems (normally electrostatic precipitators) to ensure that they meet clean air standards [89]. The design and operating conditions of electrostatic precipitators are of considerable interest in this instance because they should directly influence the properties of fly ash produced by a given power plant.

Electrostatic precipitators consist of an array of parallel plates with wires placed equidistant between the plates. A large potential difference is maintained between the wire (charging electrode, very high negative charge) and the parallel plates (collecting plates, grounded). When the flue gas exiting the boiler of a power plant is directed through an electrostatic precipitator, the particulates in the flue gas accumulate negative charge from the charging electrode and are attracted to the grounded collecting plates [18,38]. Frequent rapping of the collecting plates keeps the particulate layer from building up to a thickness that would impair precipitator performance. The particulates that are vibrated off the precipitator plates fall into a hopper (see Figure 9) and are then pneumatically transferred to a storage silo [78]. Obviously, the particulates referred to above are most commonly called fly ash.

The pertinent factors affecting the field performance of electrostatic precipitators include coal properties (i.e., heating value, sulfur content and ash content), power plant operating parameters (i.e., pulverizer performance, boiler residence time, flue gas temperature and velocity, and electrostatic precipitator field strength), and fly ash properties (i.e., ash resistivity and particle size) [18, 77, 89, 94]. The major design change needed for electrostatic precipitators used to collect particulates produced from burning western coals is mainly one of precipitator location. The

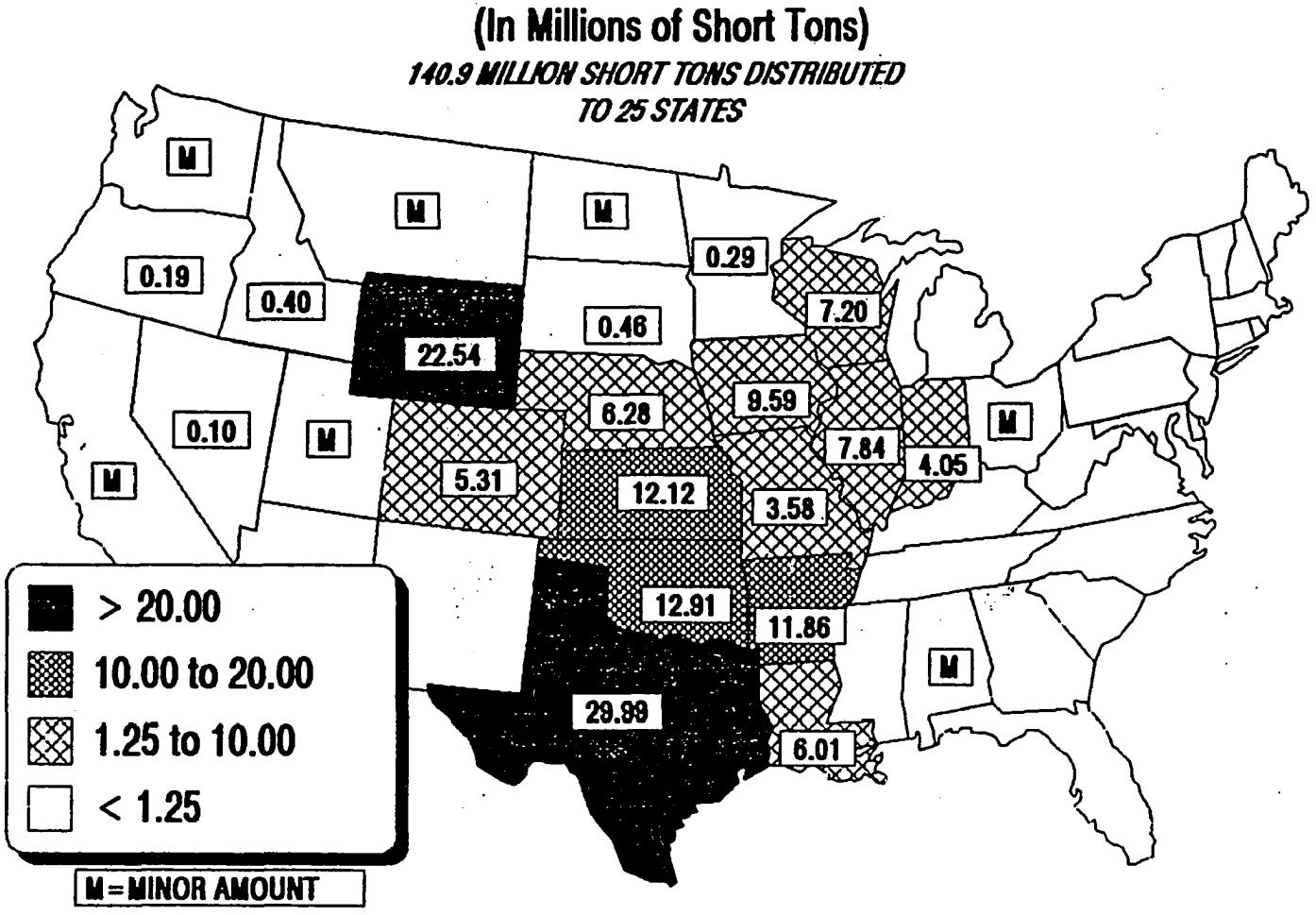


Figure 8. States utilizing coal from Wyoming (adapted from McNair as shown in [41])

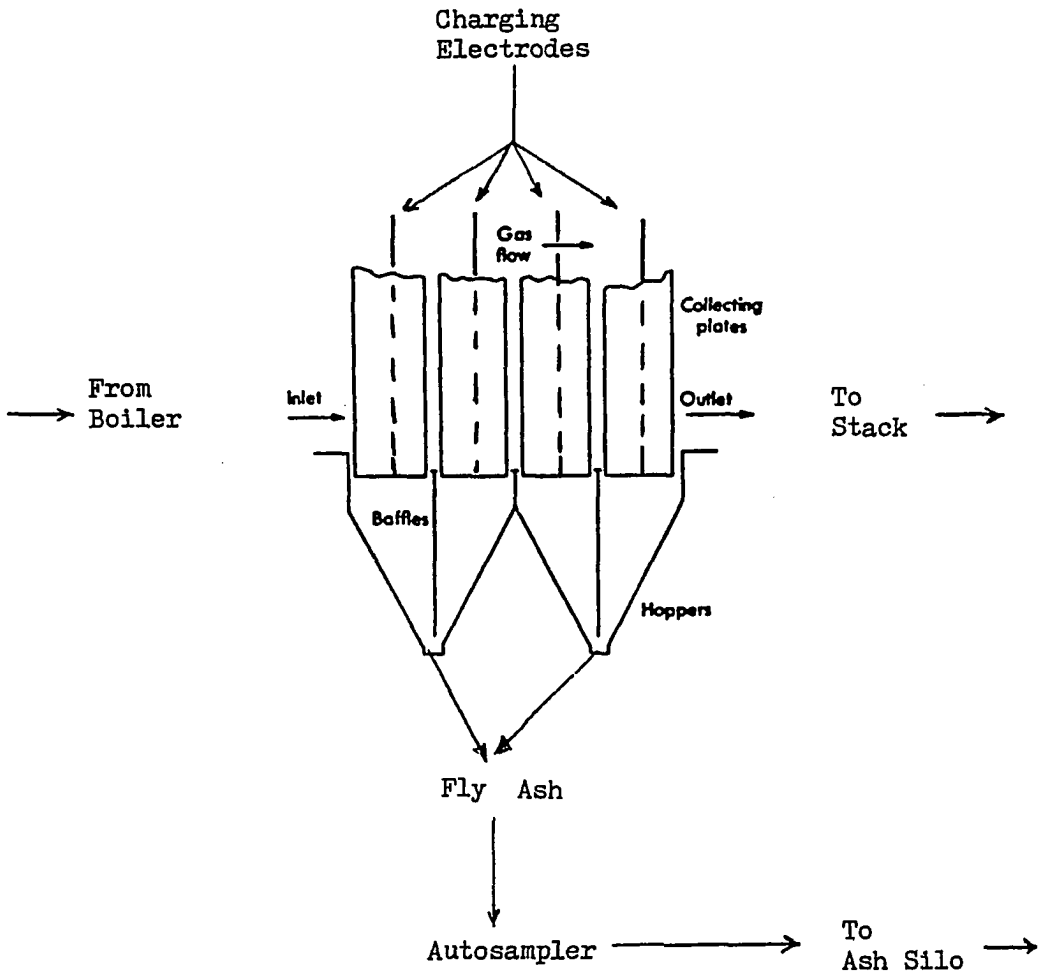


Figure 9. Generalized diagram showing the major features of an electrostatic precipitator (adapted from [78])

electrical resistivity of fly ash is dependent on both the flue gas temperature and chemical composition (especially the sulfur content of the coal and the basicity of the fly ash) as shown in Figure 10; and hence, it is common to utilize hot-side electrostatic precipitators to collect fly ashes generated form low-sulfur (western) coals. This is in contrast to the cold-side electrostatic precipitators that are normally used to collect fly ashes produced from moderate and high sulfur coals. The terms "hot-side" and "cold-side" simply refer to the location of the electrostatic precipitator relative to the primary air preheater (see Figure 11) [89, 91]. Please note from Figure 11, that the two types of electrostatic precipitators function at considerably different temperatures in order to utilize the strong relationship between fly ash resistivity and temperature.

Electrostatic precipitators are not trouble free devices — in fact, there has been a great deal of research work attempting to define additives that will help increase precipitator performance and/or increase the time intervals between precipitator maintenance periods [28]. The most common additive for cold-side electrostatic precipitators is sulfur dioxide injection into the flue gas just prior to the precipitator entrance. Hot-side electrostatic precipitators appear to suffer more maintenance problems than cold-side precipitators [91]. The most common method of increasing the efficiency of a hot-side electrostatic precipitator is to reduce the resistivity of the fly ash. This is commonly done by adding sodium bearing compounds (i.e., sodium carbonate, etc.) to the raw coal feed [28,68]. Obviously, the utilization of precipitator enhancement additives should influence the characteristics of the fly ash exiting a given power plant; however, how this may affect the engineering properties of fly ash is presently an area of dispute.

Characteristics of Fly Ash

Fly ash is a rather complex material because it is formed during the turbulent burning of pulverized coal. Many pulverized coal burning power plants have peak boiler temperatures of approximately 1500°C and the fly ash is suspended in the flue gas stream for only a brief period of time (measured in seconds) before it is removed via the particulate collection system. Hence, the

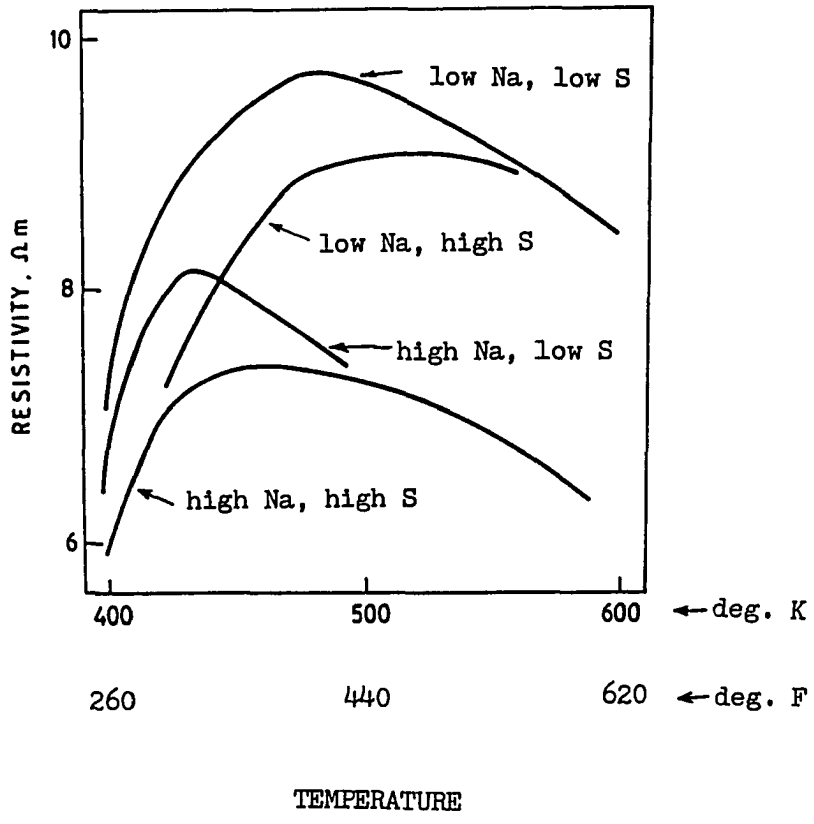


Figure 10. Electrical resistivity of coal ashes (adapted from [77])

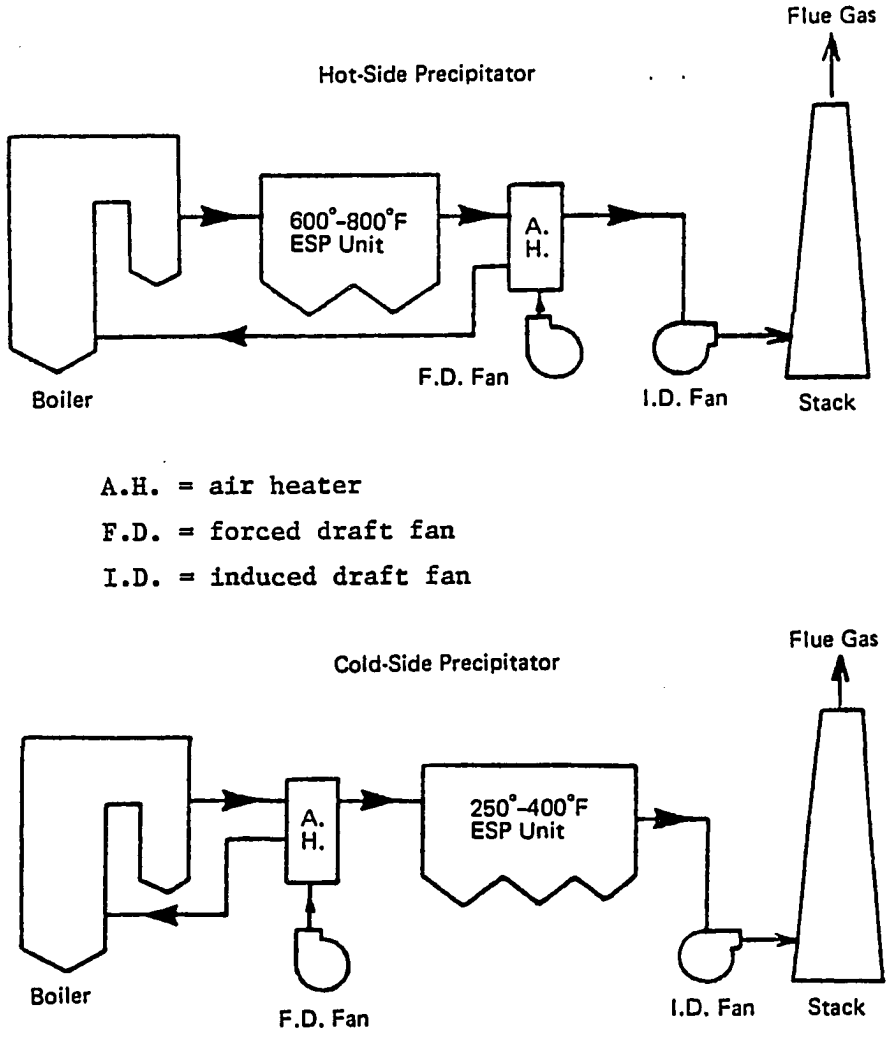


Figure 11. Diagrams of hot-side and cold-side electrostatic precipitators (adapted from [91])

thermal history of a given fly ash is generally unknown and, at best, can only be roughly estimated. The purpose of this section is to give a general overview of the morphology, chemistry and mineralogy of fly ash.

It is pertinent to add that due to the particulate nature of fly ash, such an overview can be approached in two distinctly different manners. First, a microscopic point of view could be used to describe fly ash. Such a method would utilize information obtained from individual fly ash particles. The second method, a macroscopic point of view, would utilize information obtained from bulk fly ash samples (average value of billions of individual particles). Both the microscopic and the macroscopic methods have their strengths and weaknesses; however, when both methods are employed in unison, they provide a reasonably concise description of what fly ash is. Researchers have long been aware of this fact but they have had little success in convincing industry of the potential benefits of a microscopic—macroscopic classification scheme. Hence, current specifications have adopted only the macroscopic view of fly ash. Throughout this dissertation, fly ash properties will be described using both the microscopic and the macroscopic perspectives.

Also, the reader must be informed about the difference between coal ash and fly ash. Coal ash is simply an ashed sample of coal that is prepared in a laboratory environment. Fly ash is the particulate material that is removed from the flue gases of a pulverized coal burning power plant. Many papers in the literature tend to categorize (or confuse) coal ash with fly ash; however, this is not a reasonable thing to do because a single source of coal generally produces coal ash and fly ash that have distinctly different physical and chemical properties. Hence, when reading the literature, one must be certain of which type of ash the author is referring to. This dissertation will only be concerned with the chemical and physical properties of fly ash.

Morphology

Fly ash morphology is strongly dependent on the thermal history of the coal/fly ash particles. Researchers have conducted detailed studies concerning the formation of fly ash from

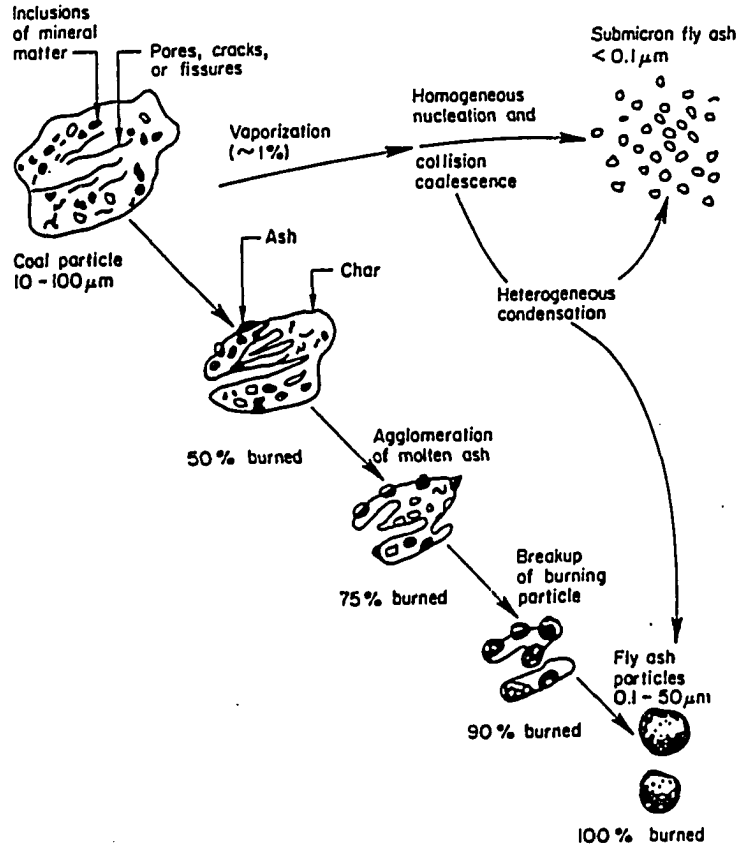
the burning of pulverized coal [77]. The process can be conceptualized as shown in Figure 12. The top half of Figure 12 illustrates the burning of an individual particle of coal. The bottom half of Figure 12 simply illustrates the processes occurring in the mineral matter inclusions while the coal is burning away and fly ash particles are forming. Often, much of the mineral matter in the coal will melt, or at least partially melt, during the trip through the boiler. Also, volatile inorganic (and organic) constituents that were vaporized in the boiler may condense, either into discrete particles or on the surfaces of fly ash particles, as the temperature decreases.

The dynamic environment described above causes fly ash to have a predominantly spherical morphology. This morphology is illustrated in Figure 13. Several morphological classification schemes have been proposed for fly ash [24, 90, 100] and all are roughly equivalent. As an example, Fisher's [24] classification scheme is shown in Figure 14. Briefly, the classification scheme simply categorizes the individual fly ash particles based on shape and opacity. Hence, this classification scheme defines 11 distinct types of particles. Obviously, particle morphology is strongly dependent on both particle size and composition. Large particles may pass through the boiler nearly unaltered, especially if they have a high melting point temperature (for example, a quartz grain). Thus, they tend to produce particles with an angular morphology (see the upper part of Figure 14).

Color is an important macroscopic property of fly ash. Most fly ashes range in color from black to white. Black colored fly ashes tend to contain a large portion of partially burned coal, they may also contain ferrite spinels (most commonly magnetite) which can contribute to their dark color. In contrast, fly ashes that range in color from buff to white, generally contain very little unburned carbon. Hence, color can be used to qualitatively assess the carbon contents of different fly ashes.

Chemistry

The bulk chemical composition of fly ashes from different power plants is quite variable. Helmuth [30] has summarized the chemical composition of fly ash as listed in the literature during



Concurrent processes in mineral matter inclusions

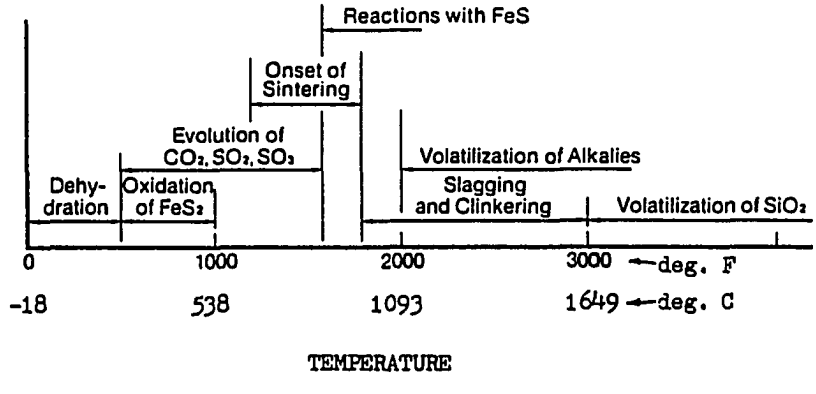


Figure 12. Idealization of the process of fly ash formation (Flanagan, as shown in Helmuth [30], adapted from [18])

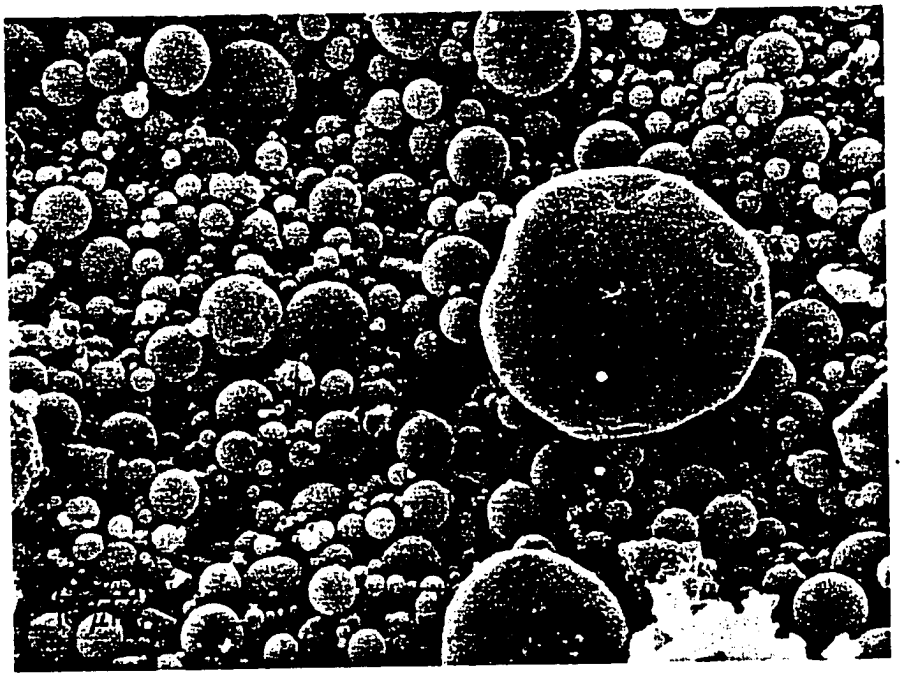


Figure 13. Scanning electron micrograph of fly ash [30]

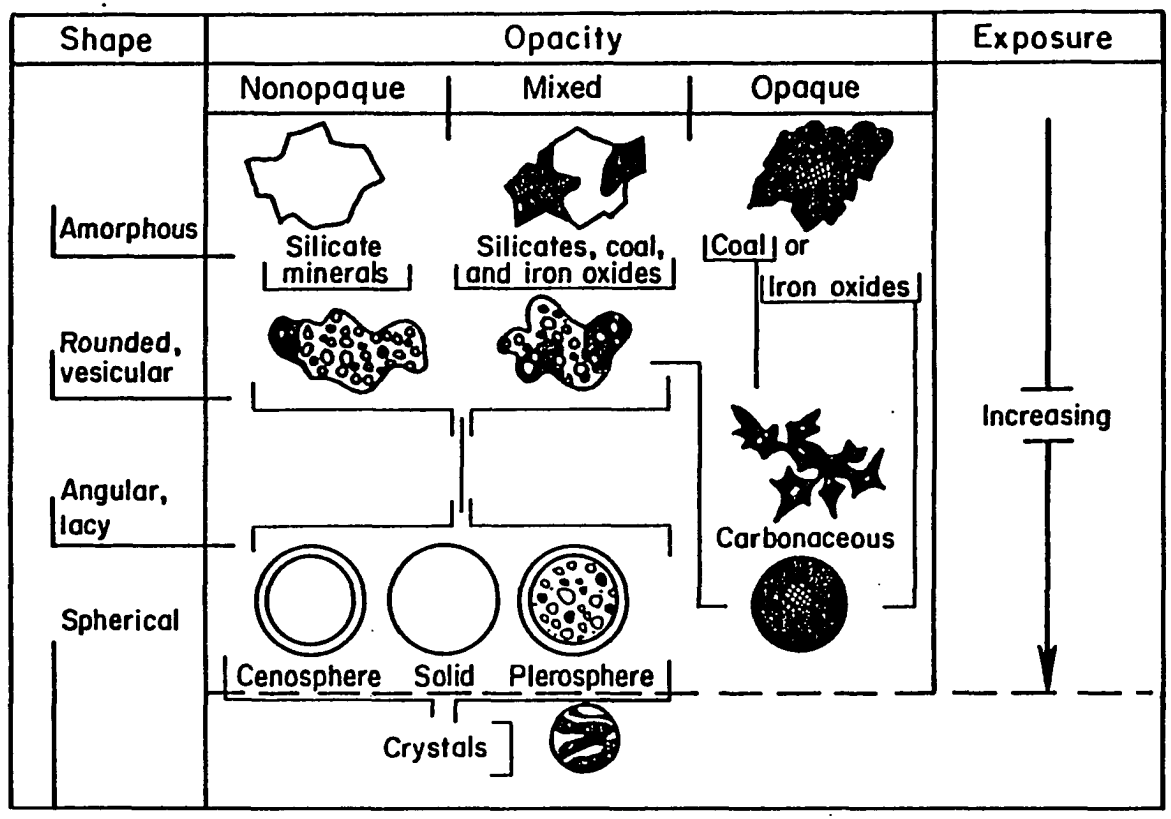


Figure 14. A morphology classification scheme for fly ash particles [24]

the 1960s, 1970s and 1980s. An updated version of Heimuth's table is given in Table 1. Fly ash assays from the late 1930s and early 1940s [19, 20], the mid-1950s [12] and the mid to late 1980s [37, 86] have been included in Table 1 to illustrate several points. Please note in Table 1, that the early studies did not list the specific rank of coal from which the fly ash was produced. However, most of the articles did list the location of the power plant that was investigated. Hence, in Table 1, a coal rank preceded by a question mark (e.g., ?Bituminous) simply indicates that the designation was made by this author and is uncertain.

The first comment that can be made about Table 1 is that it is quite difficult to define the composition of a "typical" fly ash. The chemical composition of fly ash is dependent on both the parent coal and the power plant design and operating conditions (as described earlier in this dissertation). However, it is possible to identify which elements need to be measured and to define a very rough range of concentration for these elements (see Table 2). Such information is very important for analysts because it may dominate the selection of the method used for quantifying the various elements.

The second comment that can be made about Table 1 concerns the lack of interest of researchers in measuring all the elements present in a given fly ash (compare Tables 1 and 2). This stems from the fact that the analytical methods used to quantify fly ash were adopted from existing standard analytical (gravimetric) methods used for portland cement. It is not unusual to use standard analytical methods to quantify vastly different materials; most standard methods perform well when properly applied and accurately interpreted. In fact, most of the gravimetric methods used for the analysis of portland cement were almost surely adopted from the classical techniques originally designed for the analysis of rocks [44]. However, the point of this argument is that all of the major and minor elements (i.e., elements with concentrations greater than 0.1%) must be determined to adequately correct for systematic errors in gravimetric analyses. The determination of Al (expressed as Al_2O_3) in fly ash is a good example of a systematic error that currently exists in

Table 1. Ranges of composition of fly ashes as listed in the literature (Jarriage and Sersale adapted from Helmuth [30])

	Source:	Davis 1937, 41	Brink & Halstead 1956	Jarriage 1971	Sersale 1980	Schlorholtz 1983-87	Joshi 1987
Component	Coal:	?Bituminous	?Bituminous	Lignite	Bituminous	Sub-bituminous	Sub-bituminous
SiO_2		28-51	33-52	18-25	50	28-41	55-59
Al_2O_3		16-31	15-28	12-15	30	15-20	20-23
Fe_2O_3		4-19	8-31	6-8	7	4.5-7.5	3-7
CaO		1-11	1-12	43-49	2	22-32	6-10
MgO		0-2	0.7-1.4	2-3	5	4-7	1-2
Na_2O		0-2	0-2	a	a	1-4.5	0-3
K_2O		1-3	1-3	5	5	0.2-0.6	0.6-1
SO_3		0-3	0-3	5-9	n/mb	1-5	0-0.5
TiO_2		0.5-1.5	inc[c]	n/m	n/m	0.5-1.5	0.9-1.8
P_2O_5		inc	inc	n/m	n/m	0.5-2.5	n/m
SrO		n/m	n/m	n/m	n/m	0.4-0.5	n/m
BaO		n/m	n/m	n/m	n/m	0.6-1.0	n/m
C		1-30	0-16	1-3	1-6	n/m	n/m
LOI		1-32	1-18	1	5-12	0.1-0.9	0.3-1

[a] indicates that the sodium and potassium are expressed as a single number.

[b] n/m = not measured.

[c] inc = included in Al_2O_3 determination.

Table 2. Typical ranges of composition for many fly ashes

Component	Range of Concentration (wt. %)
SiO_2	20 - 60
Al_2O_3	10 - 40
Fe_2O_3	2 - 50
CaO	0 - 50
MgO	0 - 10
SO_3	0 - 10
Na_2O	0 - 6
K_2O	0 - 4
P_2O_5	0 - 3
TiO_2	0 - 2
BaO	0 - 1.5
SrO	0 - 1
Loss on ignition (LOI)	0 - 30

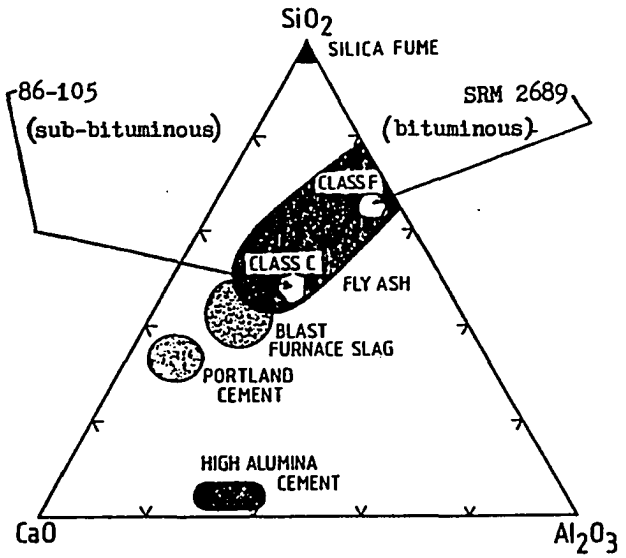
the standard fly ash analytical methods [5]. The Ti and P present in a fly ash sample tend to accumulate in the R_2O_3 group during the normal separation process, and this leads to a systematic error in the gravimetric Al_2O_3 determination [4, 43]. This error can be corrected quite easily but, at present, it has simply been ignored [85]. Hence, one must be careful when interpreting the chemical information listed in Table 1. When the TiO_2 and P_2O_5 contents are not specifically listed in Table 1 then they have probably been incorporated into the Al_2O_3 determination.

Finally, it is apparent from Table 1 that there is a very rough relationship between coal rank and the chemical composition of fly ash. This is the reason for the classification scheme that is currently used to specify fly ash for use in portland cement concrete [5]. The details of the classification scheme will be described later in this dissertation.

On a microscopic scale, fly ash is extremely heterogeneous. Recent studies [70, 76, 92] have indicated that individual fly ash particles may have chemistries that are drastically different from the bulk chemical composition of the ash. This fact is illustrated in Figure 15 using information extracted from Stevenson and Huber [92] and Glasser, Diamond and Roy [27]. The bulk chemistries of the two fly ash samples are also listed in the figure. This emphasizes the large difference between bulk chemistry and individual particle chemistry.

Due to the microscopic heterogeneity of fly ash, many researchers have utilized additional techniques to enhance their ability to study the partitioning of elements (and/or minerals) in fly ash. The major techniques that have been employed are density separations [31, 33, 100], particle size separations [24, 25, 31, 78] and leaching of specific components using acids and/or other organic and inorganic solvents [10, 42, 81]. Magnetic separation is also useful in some instances because it can be used in combination with the previously mentioned techniques to obtain a more specific separation. However, one must be careful in generalizing the results obtained from the various studies because no standardized experimental methods have been used by any of the researchers. For example, special care should be taken when interpreting the particle size studies

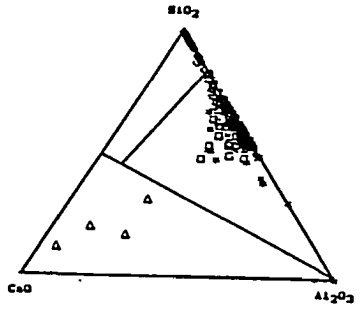
Macroscopic Perspective
(bulk chemistry)



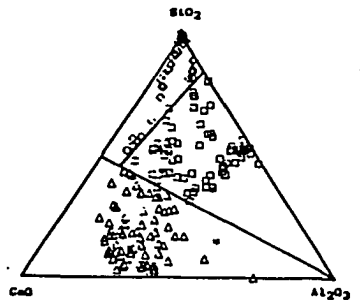
Bulk Assays (wt.%)

Sample	SRM 2689	86-105
SiO_2	51.5	34.1
Al_2O_3	24.4	18.3
Fe_2O_3	13.3	5.9
CaO	3.0	26.0
MgO	1.0	5.0
Na_2O	0.3	2.7
K_2O	2.6	0.3
SO_3	0.70	3.12
LOI	1.80	0.41
Moisture		0.05
Total	98.6	95.9

Microscopic Perspective
(particle chemistry)



SRM 2689



86-105

Figure 15. **An illustration of fly ash heterogeniety on a microscopic basis [27,92]**

that utilized air classification for size-separation [24, 31], because any particle separation method utilizing aerodynamic properties of a given material will be biased due to density and shape differences between different particles [48]. Hence, the information should only be utilized in a qualitative manner until the reliability of the techniques have been verified by several different laboratories.

Several researchers have also investigated the variability of the bulk chemistry of fly ash from individual power plants as a function of time (i.e., sampling date) [35, 36, 56, 71, 72]. Such studies are pertinent to the effective utilization (or disposal) of fly ash because without this information, it would be difficult to ascertain the chemical consistency of a given source of fly ash. If a fly ash marketing agent cannot convince potential users of his ability to provide a uniform product, then to state it bluntly, he will not have any customers.

In general, most of the research has indicated that the concentration of the major and minor elements (usually reported as oxides) in fly ash were reasonably constant as a function of time. A summary of the various test results for high-calcium fly ashes are listed in Table 3. The study by Holcombe et al. [35] was perhaps the most scientifically designed fly ash variability monitoring experiment to date; however, it was not included in Table 3 for two reasons. First, the study did not explicitly state the number of samples that were analyzed during the investigation. And second, the ash studied by the researchers was taken from a fly ash—water sluice line rather than from a dry storage hopper. Hence, the ash had already been leached (to some unknown extent) by the water. The paper did not list any explicit details about correcting for the water soluble elements initially present in the bulk fly ash.

Mineralogy

Fly ash is an intimate mixture of both crystalline and non-crystalline (glassy) material. To date, many studies have addressed the identification and quantification of the crystalline compounds (i.e., minerals) present in a wide variety of fly ashes. However, the investigation of the

Table 3. Variability of the chemical composition of high-calcium fly ashes

Source:	McKerall and Ledbetter			Pitt et al.		Schlorholtz et al.		
Sampling date:	unknown	unknown	unknown	1981, 1982	1981, 1982	1983	1984	1985
Fly Ash designation:	D	H	W	Neal 4	Council Bluffs	Ottumwa	Ottumwa	Ottumwa
Number of samples:	16	7	7	34	28	8	17	17
Component	CV(%)a	CV(%)	CV(%)	CV(%)	CV(%)	CV(%)	CV(%)	CV(%)
SiO_2	7.4	4.6	3.7	9.0	5.3	4.3	4.0	5.1
Al_2O_3	7.4	8.2	6.5	1.8	2.3	2.0	1.9	1.8
Fe_2O_3	15.5	7.7	3.9	7.9	5.2	2.7	3.1	7.4
CaO	13.8	5.4	8.7	14.6	4.5	2.8	2.9	2.2
MgO	27.2	11.2	12.7	18.5	10.7	3.8	3.5	2.6
SO3	24.8	9.0	16.8	–[b]	–	13.2	17.1	17.2
Na2O	–	–	–	23.9	6.5	12.2	6.2	24.4
K2O	–	–	–	58.8	12.2	7.5	10.0	7.9
TiO2	–	–	–	10.1	7.3	3.4	3.6	2.8
P2O5	–	–	–	–	–	21.3	12.8	23.9
LOI	68.4	20.0	25.0	–	–	20.8	19.2	24.0

aCV = coefficient of variation = (standard deviation ÷ mean) × 100.

[b]No data.

glass fraction of fly ash has lagged behind the investigation of its crystalline fraction. This is largely due to the fact that the glass fraction of fly ash is normally studied on a microscopic basis, whereas the crystalline fraction can be studied in bulk samples via x-ray diffraction. Hence, the study of glass fraction of fly ash has been painstakingly slow due to the extreme heterogeneity that fly ash exhibits on a microscopic scale.

The mineralogy of fly ash is most commonly studied using x-ray diffraction (XRD) techniques [52, 64, 90, 100] or optical microscopy [11, 100]. Recent studies have also utilized spectroscopic techniques (i.e., infrared analysis [23], microfocus Raman analysis [82], gama-ray spectroscopy and x-ray absorption spectroscopy [39]) and electron microscopy (both scanning and transmission techniques, plus electron diffraction [74, 75, 76]) to probe deeper into the structure of fly ash. Complementary analytical methods, such as differential thermal analysis, or thermogravimetric analysis, have also been employed. Hemmings [32] gives an excellent overview of how these various techniques have been applied to fly ash research. Also, the whole suite of chemical and physical separation techniques that were employed to enhance the elemental characterization of fly ash have also been used to enhance the mineralogical study of fly ash. Hence, although much characterization work remains, the past ten years have contributed greatly to our present understanding of the fundamental constituents of fly ash.

The crystalline compounds that are normally found in fly ash are summarized in Table 4 [22, 52, 55, 64]. It is pertinent to add that only four of the compounds, namely quartz, mullite, hematite and a ferrite spinel, are commonly found in fly ashes obtained from the burning of Bituminous coal. All of the remaining compounds have been identified in fly ashes produced from sub-bituminous or lignitic coals. Hence, it is apparent that the mineralogy of fly ashes produced from Bituminous coals is considerably less complex than that of Western coals. Also, Table 4 only lists the primary minerals normally found in fly ashes, secondary phases, that often form from the interaction of bulk fly ash with the environment (i.e., water), will be discussed in another section of this dissertation.

Table 4. Crystalline compounds commonly found in fly ash

Mineral Name	General Composition	Range of Concentration Commonly Found in Fly Ashes	Commonly Found in Iowa Fly Ashes
Quartz, low	SiO_2	3 - 15	Yes
Mullite	$Al_6Si_2O_{13}$	0 - 25	Yes
Hematite	Fe_2O_3	0 - 5	Yes
Ferrite Spinel	$(Mg,Fe)(Fe,Al)_2O_4$	0 - 20	Yes(~Fe_3O_4)
Periclase	MgO	0 - 5	Yes
Lime	CaO	0 - 10	Yes
Anhydrite	$CaSO_4$	0 - 5	Yes
Tricalcium Aluminate	$Ca_3Al_2O_6$	0 - 10	Yes
Melilite structure	$(Ca,Na)_2(Mg,Al,Fe)(Si,Al_2)O_7$	0 - 2	rare (~$Ca_2Al_2SiO_7$)
Merwinite	$Ca_3Mg(SiO_4)_2$	0 - 8	Yes
Sodalite structure	$Ca_2(Ca,Na)_6(Al_1Si)_{12}O_{24}(SO_4)_{1-2}$	0 - 3	Yes (~ $Ca_4Al_6O_{12}(SO_4)$)
Sodium sulfate	Na_2SO_4	?	No

Several researchers [53, 58, 64, 79] have made quantitative estimates of the amount of the various crystalline compounds present in fly ash. Quantitative x-ray diffraction (QXRD) analysis is the analytical technique that is commonly used to measure the compounds in fly ash. Most researchers have employed the reference intensity ratio method to calculate the concentration of a given compound from its measured diffracted intensity. This method has several distinct limitations which have been explained in detail by McCarthy et al. [52, 53]; and hence, for a material as complex as fly ash, one must be aware that the results are only semi-quantitative at this time. The range of concentration of the various compounds measured via QXRD analysis for a wide variety of fly ashes is summarized in Table 4.

The QXRD results indicate that only about half (more often considerably less) of a given fly ash is composed of crystalline compounds. The remaining portion of fly ash is amorphous to x-rays and thus, x-ray diffraction techniques yield little distinct information about how the various elements are combined (bonded) in the solid material. X-ray diffraction analysis does suggest, however, that more than one type of glass may be present in fly ash.

An example of the information that is obtained from the diffraction studies of glass, sodium silicate glass specimens in this instance, is illustrated in Figure 16. The trend indicates a shift in the location of the glass scattering halo as a function of chemical composition. Diamond [21] was the first to recognize that the glass scattering halo, which is commonly observed in x-ray diffractograms of fly ash, appeared to be dependent on the bulk analytical calcium oxide content of the fly ash. This trend is illustrated in Figure 17a. Mehta [58] observed a similar trend and McCarthy, who had access to a much larger population of fly ashes of widely different chemical compositions, re-evaluated the trend between glass scattering maximum and bulk calcium content in fly ash (see Figure 17b) [32]. The exact meaning of the observed trend is still a matter of dispute; however, most researchers are in general agreement that the trend indicates a decrease in the polymerization of the silicate glass network.

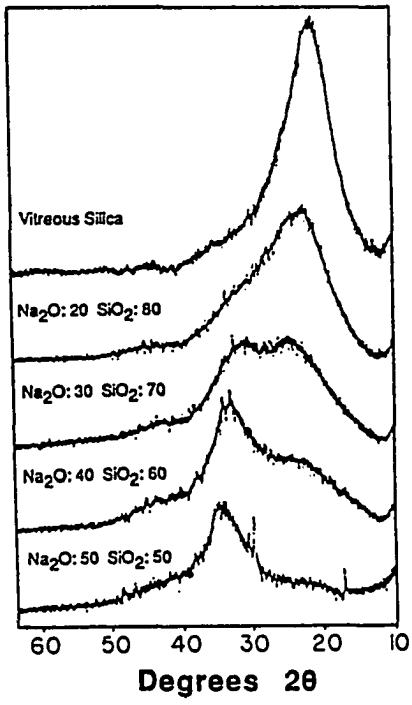


Figure 16. X-ray diffraction patterns for glasses [32]

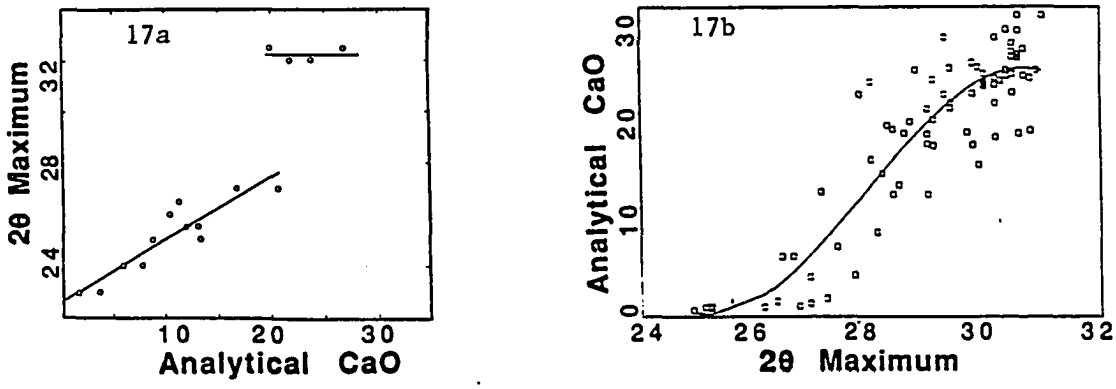


Figure 17. Relationships between bulk analytical CaO content and the location of the glass scattering halo in fly ash [32]

Hemmings and Berry [32] have proposed that fly ashes contain two glass types which are composed of different structural units (see Figure 18). The first glass type is nearly a 3D random network that is composed mostly of silica. The second glass type is distinctly less polymerized because of the large number of modifier cations (Ca, Mg, Na, K, etc.) present in the glass. Recent information obtained from infrared and microfocus laser Raman spectroscopies, tend to reinforce this hypothesis [32]. However, one must be very careful when interpreting the information obtained from x-ray diffraction and infrared spectroscopy because these methods only yield bulk (i.e., average) trends. One must remember that fly ash is so very heterogeneous on a microscopic scale that a wide range of glass compositions appear to be possible.

Bergeson [8] has made a detailed study of the bulk glass portion of several Iowa fly ashes. The major tools employed in his study were x-ray diffraction (both qualitative and quantitative routines) and x-ray fluorescence spectrometry for elemental analysis. Bergeson's work indicated two important aspects of Iowa fly ashes. First, his work clearly indicated that the glass portion of most of the fly ashes produced in Iowa was derived from a smectite-like clay source present in the raw coal. And secondly, his work indicated a rather good correlation between the $(CaO+MgO)/(SiO_2+Al_2O_3)$ ratio of the glass and the same ratio calculated from bulk elemental analysis (correlation coefficient, R, = 0.991; based on 12 data points). Hence, Bergeson concluded that bulk chemistry could be utilized to calculate a "reactivity index" for the glass portion of Iowa fly ashes. The reactivity index (RI) was simply defined as the ratio of network modifiers to network formers present in the glass fraction of a fly ash. Bergeson's study did not include Na, K, Sr or Ba as modifiers nor did it include P as a potential network former, however, these elements are often only minor constituents of many fly ashes.

Other researchers [74, 76] have published data that are in general agreement with the "reactivity index" trend reported by Bergeson. The composite trend is shown in Figure 19. It is pertinent to mention that Bergeson calculated his "reactivity index" by subtracting the composition

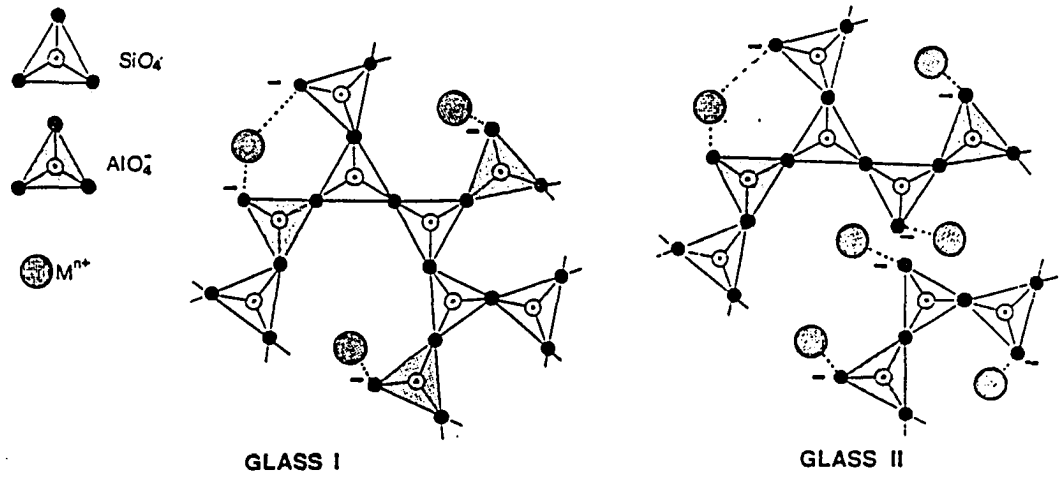


Figure 18. The proposed structural differences between fly ash glass types [32]

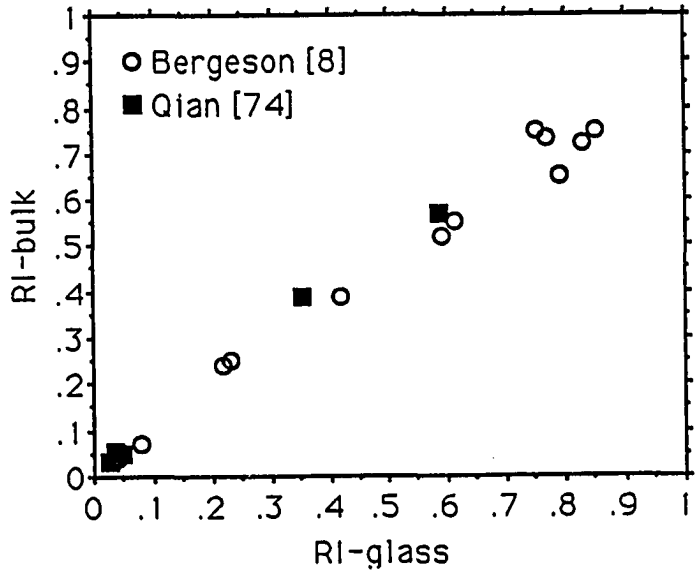
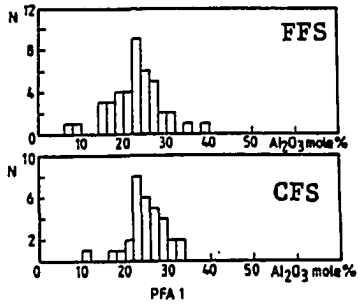


Figure 19. The relationship between the reactivity index (RI) calculated from bulk chemistry versus that measured in the glass fraction

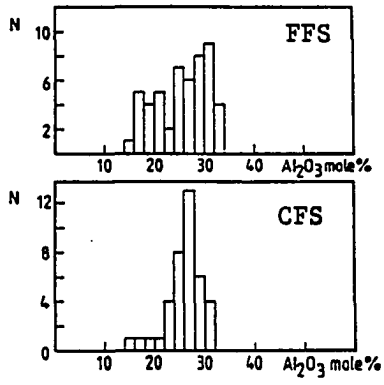
of the crystalline compounds (determined via x-ray diffraction) from the bulk chemical assay (determined via x-ray fluorescence). Qian et al. [74, 76] performed direct quantitative analyses on numerous glassy areas in fly ashes by using analytical electron microscopy techniques. Thus the trend indicated in Figure 19 represents information gathered from two totally different analytical techniques. At present, it is uncertain if the incorporation of additional elements into the "reactivity index" calculation would improve the correlation between the bulk composition of the glass and the bulk chemical assay.

The microstructure of the glass present in several fly ash samples has been investigated in detail by Qian and his associates [74, 75, 76]. Fly ash samples were studied using analytical electron microscopy and high resolution transmission electron microscopy coupled with selected area diffraction techniques. The majority of the work concentrated on fly ashes produced from Bituminous coals (i.e., low-calcium fly ashes); only two of the fly ashes had Ca contents greater than five percent. In general, their research indicated that glass composition can change significantly over relatively small distances (say roughly 1 to 2 microns). Figure 20 illustrates the variability they observed for the Al content of the glass portion of their fly ashes. The bulk chemical assays of these fly ashes are summarized in Table 5. It is interesting to note in Figure 20, that the high-calcium fly ash exhibits the greatest variability in Al content. Again, this is a good illustration of the extreme heterogeneity of fly ash. Qian and his associates [75] have constructed a table which briefly summarizes their experimental findings; this is summarized in Figure 21, along with a diagram to illustrate the concept of microscopic fly ash heterogeneity.

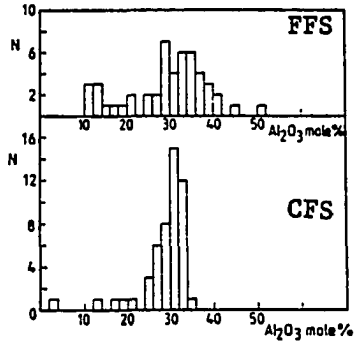
Few researchers have had the opportunity to routinely monitor the mineralogy of a single source of fly ash for long periods of time. Typically, the laboratories that receive a large number of fly ash samples from a single source are only testing laboratories, rather than research laboratories. Testing laboratories do not normally have access to the equipment needed to probe the mineralogy of fly ash (x-ray diffraction, etc.). They only monitor a few of the physical properties of fly



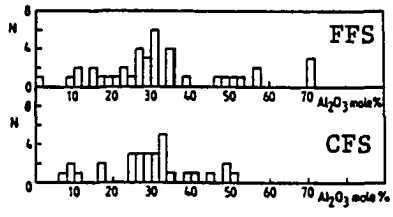
PFA 1



PFA 3



PFA 5



PFA 6

N = number of observations, typically about 30 to 50

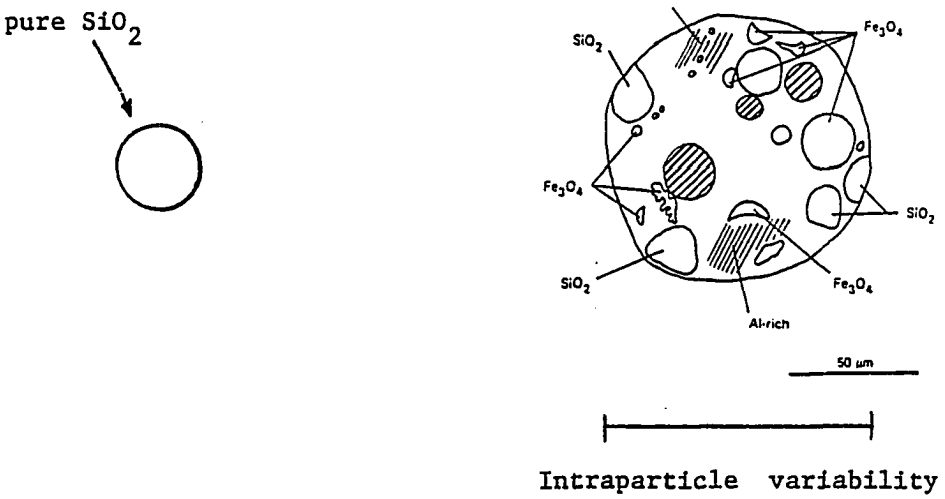
FFS = fine focal spot, less than 0.1 microns in diameter

CFS = coarse focal spot, about 0.5 to 1 microns in diameter

Figure 20. Concentration of Al_2O_3 in the glass fraction of various fly ashes [74]

Table 5. Bulk chemical assays of the fly ashes investigated by Qian and Glasser [74]

	Sample			
Oxide (wt. %)	PFA 1	PFA 3	PFA 5	PFA 6
SiO_2	51.69	48.12	49.89	40.28
Al_2O_3	30.37	31.17	38.51	24.86
CaO	1.38	2.68	2.83	21.32
K_2O	4.28	4.07	1.34	0.89
Na_2O	1.19	0.78	0.24	1.84
Fe_2O_3	9.10	10.85	5.38	5.38
SO_3	0.54	0.72	0.46	1.48
MgO	1.44	1.61	1.34	3.96



Chemical Variability in Fly Ash Glasses

Approximate Scale		
mm	μm	nm
Interparticle Variability---		
	---Intraparticle Variability---	
	Melting inhomogeneities: both *inter-* and *intra-*particulate; former gives rise to diffuse gradients	Classical amorphous phase separation with sharp interfaces and spinodal decomposition with diffuse interfaces

Figure 21. An illustration of intraparticle variability versus interparticle variability in fly ash glasses (adapted from [74])

ash. Conversely, few research laboratories have access to the number of fly ash samples that a testing laboratory does; and hence, the current knowledge of the long-term variability of the mineralogy of fly ash is very limited. Schlorholtz, Bergeson and Demirel [86, 87] have begun to address this problem from an engineering perspective. Their research indicated that the major minerals identified in Iowa fly ashes were very consistent; however, the quantities of the various minerals appeared (qualitative x-ray diffraction measurements only) to be changing significantly. Quantitative x-ray diffraction studies were planned to supplement the initial research findings.

Few researchers have attempted to relate fly ash mineralogy to bulk fly ash chemistry. Again, research has been stymied by both a lack of interest and, more importantly, a lack of reliable information. McCarthy et al. [54] have attempted to correlate the mineralogy and chemistry of western fly ashes but, at the present time, the results of the correlation are only qualitative. Theoretical studies [49, 96] have made progress but only in a very limited sense because of the gross nonequilibrium processes that normally influence the formation of fly ash.

Properties of Fly Ash - Water Mixtures

The physical and chemical properties of fly ash-water mixtures were largely ignored until the late 1970s, when fly ash from western coals became more common. Early researchers [1, 12, 19, 20, 65], who studied Bituminous coal (low-calcium) fly ashes, found that only a small fraction of any given fly ash was soluble in water. No mention was made of any setting or hardening reactions that were observed in these low-calcium fly ashes. Hence, researchers investigated the use of chemical activators, such as portland cement, lime and calcium hydroxide, to initiate the cementitious nature of these fly ashes. In contrast, many of the fly ashes produced from the burning western coal (high-calcium fly ashes) are quite reactive with water. Typically, the reactivity of these western coal fly ashes is grossly related to their greatly increased analytical CaO content. Many of these high-calcium fly ashes are designated as "self-cementitious" because they solidify (set) and harden when mixed with water. The purpose of his section is to describe the basic properties of self-

cementitious fly ashes, how such properties are measured, and current hypotheses concerning the chemical reactions that occur in fly ash-water mixtures (i.e., fly ash pastes).

The pertinent engineering properties of cementitious materials that are normally measured are: (1) setting behavior; (2) heat evolution characteristics; (3) volume stability; and (4) strength. Often a fifth characteristic, namely durability, is also included in the list. However, the durability of fly ash pastes, mortars or concretes will not be discussed in this dissertation.

The setting behavior of cementitious materials is very important in field applications because unpredictable setting behavior can lead to the total disruption of a construction project. The term "time of set" is a rather arbitrary term. It simply refers to the gradual change of a cement-water mixture from its initial (i.e., fluid) state, to a semi-solid (called initial set), and then to a solid (called final set). Many researchers have employed different experimental techniques to measure time of set in portland cement pastes (see ASTM C 191 and C 266 in [4]). However, many high-calcium fly ashes set and harden so fast that the standard test methods commonly used for cements are simply not applicable. Research by Bergeson [8] has indicated that a pocket penetrometer, a common soil engineering testing device, is quite useful in monitoring the time of set of fly ash pastes. All of the test methods mentioned above are based on the same concept, namely a deformation-time relationship, and each method is equally correct as long as one realizes that all measurements are relative rather than absolute.

The heat evolution characteristics of cementitious materials are very important in the design and construction of massive structures. Since most cementitious materials have very low coefficients of thermal diffusivity, the heat produced from the exothermic hydration reactions may accumulate in the structure, and hence, thermal stresses could potentially destroy the integrity of the structure. Descriptions of the various test methods normally used to measure the heat evolution behavior of portland cement can be found in most general cement-materials text books [46, 62]. The heat evolution of fly ashes may also be studied in a similar manner. However, many

researchers have utilized less rigorous test methods, such as constructing simple calorimeters out of expanded polystyrene foam [93] or DeWar flasks [63, 71, 87], in an attempt to better simulate field conditions, rather than to obtain strict scientific information. A recent study has employed a computer controlled adiabatic calorimeter to make detailed measurements of fly ash paste and fly ash-cement paste hydration reactions [40]. in general, most of the studies yield very similar results and they indicate that the temperature rise in fly ash pastes may exceed 50 degrees Celsius. Hence, monoliths made of fly ash may be susceptible to significant thermal stresses.

The volume stability properties of cementitious materials are fundamental to their use as construction materials. Most design calculations for concrete construction consider all structural elements as static entities. Excessive deformation, whether volume increase or decrease, can not normally be tolerated because of the brittle nature of most inorganic cementitious materials. The volume stability of portland cements are commonly assessed by monitoring the linear expansion of prismatic specimens as described in ASTM 157 [5]. Specimens can be tested as pastes, mortars or concretes. The specimens may also be subjected to various environmental conditions, such as moist or dry curing, sulfate bearing solutions or elevated curing temperatures, to better simulate design conditions.

The compressive strength of cementitious materials is the most common test that is routinely used for the design of structural elements. Compressive strength is normally measured by loading a specimen in unconfined compression until it fractures. The compressive strength of portland cement is normally tested using mortar cube specimens (see ASTM C 109 [5]) for laboratory applications, or concrete cylinders (see ASTM C 39 [5]) for field applications. However, these test procedures do not appear to be applicable to the testing of fly ash. The rapid setting and hardening characteristics of high-calcium fly ashes make it difficult to incorporate an aggregate into the test mixture. There are two basic reasons for this discrepancy. First, it is very difficult to uniformly disperse the aggregate in the fly ash paste because the total mixing time must be

drastically reduced to avoid the stiffening (and hardening) of the mixture. Secondly, the normal mortar or concrete compaction methods described in the specifications, are too time consuming and the specimens tend to stiffen during compaction. This makes it very difficult to produce test specimens with repeatable compressive strengths. Hence, when working with high-calcium fly ashes it is desirable to: (1) work with fly ash pastes; (2) to increase the water content of the mixture and decrease the mixing time to avoid premature stiffing problems; and (3) to use vibratory compaction to expel air voids and other defects from the test specimens.

The chemical and physical processes that occur when high-calcium fly ash is mixed with water are only poorly understood. To date, most research has been directed at defining the engineering properties of fly ash; little work has been conducted that addresses the fundamental aspects of fly ash. Hence, little information exists in the literature concerning the hydration reactions that occur in high-calcium fly ashes.

Several researchers have probed the mineralogy of high-calcium fly ash pastes as a function of time [3, 8, 45, 61, 63, 72]. The major hydration products that were identified by the various studies were quite similar and they are summarized in Table 6. Mings [63] conducted a study of the formation of hydration products in Iowa high-calcium fly ashes using x-ray diffraction and scanning electron microscopy. He concluded that the major hydration products were ettringite, monosulfoaluminate and straetlingite. Studies by Bergeson [8] and Kuzyk [45] later verified these findings.

Utilization of Fly Ash

To be effectively utilized a material must have specific properties that can be efficiently and economically monitored. These properties are then used to devise specifications that govern the use of the material in specific applications. Ideally, the properties that are monitored should be indicative of how the material will perform in field situations, because this allows engineers to design products with confidence. However, this ideal situation is seldom experienced. Most frequently,

Table 6. Hydration products commonly identified in high-calcium fly ash pastes

Mineral Name	Chemical formula
Ettringite	$Ca_6Al_2(SO_4)_3(OH)_{12}\cdot 26H_2O$
Monosulfoaluminate	$Ca_3Al_2O_6\cdot CaSO_4\cdot 13H_2O$
Straetlingite	$Ca_2Al_2SiO_7\cdot 8H_2O$
Portlandite	$Ca(OH)_2$
Calcium silicate hydrate	$(CaO)_xSiO_2\cdot ZH_2O$

especially in the case of by-products, the characterization stage can be rather expensive because of heterogeneity. Also, the relationship between a materials specified properties (commonly measured in a laboratory) and its actual field performance may be quite complex. This is currently the case for fly ash. Simple correlations between the laboratory tests specified for fly ash and its performance in field applications have been pursued for roughly fifty years [1, 12, 17, 19, 20, 29, 30, 58, 66], and to date they have not been concisely defined. Since the concrete construction industry is by far the major consumer of fly ash it has played a very significant role in constructing fly ash specifications. The purpose of this section is to briefly present the current ASTM specifications that govern the use of fly ash in the United States.

The current specification governing the utilization of fly ash in portland cement concrete is ASTM C 618 [5]. This specification lists the physical and chemical requirements that a fly ash must meet when it is used as a mineral admixture in portland cement concrete. The performance criteria that are currently (1988) specified for fly ash are listed in Table 7. Please note in Table 7, that the specification classifies fly ashes into two distinct groups, Class F and Class C. This classification is based on coal rank. The sampling and testing procedures commonly used for fly ash are also briefly mentioned in ASTM C 618. However, one must refer to another specification, namely ASTM C 311 [5], for a detailed explanation of the sampling and testing procedures.

Fly ash is not merely used as a filler in concrete or road base construction; rather it is used as a pozzolan. The ASTM definition of a pozzolan is as follows [5]:

> Pozzolans - siliceous or siliceous and aluminous materials which in themselves possess little or no cementitious value but will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide at ordinary temperatures to form compounds possessing cementitious properties.

Hence, current specifications assume that fly ash will be used with an activator (i.e., portland cement, etc.) of some type. Only a very brief statement in the specification alludes to the fact that many Class C (high-calcium) fly ashes exhibit self-cementitious properties.

Table 7. ASTM chemical and physical requirements for fly ash

	Mineral Admixture Class F	Class C
ASTM C 618 Chemical Requirements		
$SiO_2 + Al_2O_3 + Fe_2O_3$, min., %	70.0	50.0
SO_3, max., %	5.0	5.0
Moisture content, max., %	3.0	3.0
Loss on Ignition, max., %	6.0	6.0
ASTM C 618 Physical Requirements		
Fineness, % retained on #325 sieve	34	34
Pozzolanic Activity Index:		
With portland cement, @ 28 days, min., % control	75	75
With lime, @ 7 days, min. psi	800	—
Water requirement, max., % control	105	105
Soundness:		
Autoclave expansion or contraction, max., %	0.8	0.8
Uniformity Requirements:		
Specific gravity, max. variation from avg., %	5	5
Fineness, max. variation from avg., %	5	5

Since their adoption in 1954, the ASTM specifications for fly ash have been subject to nearly continuous revision. Mielenz [60] gives an excellent discussion of the historical development of the various specifications for fly ash. The exact reason for the continuous alteration of the fly ash specifications is difficult to pin point; however, the major reason that is often mentioned in the literature is that the specifications are too restrictive (i.e., prescriptive) [14, 50, 51, 56, 57, 59, 60, 80]. Many users and marketing agencies believe that the fly ash specifications should be based solely on performance specifications. They also think that the current chemical specifications should be discarded. However, few of the critics can agree on the exact type of performance tests that should be specified for fly ash.

EXPERIMENTAL METHODS

Research Approach

Background

Research conducted under project HR-225 lead to the knowledge that the elemental composition of Iowa fly ashes remained relatively consistent over time [72]. These results verified the findings of an earlier in-house Iowa Department of Transportation (IDOT) research project on fly ash variability [36]. However, the in-house IDOT research project also indicated that the compressive strength of fly ash mortar cube specimens exhibited extreme variability as a function of sampling date. Isenberger suggested that further work should be done to confirm the observations because the experimental methodology was subject to "a significant amount of operator variability" [36]. Hence, IDOT personnel continued molding water-sand-fly ash mortar cubes in accordance to Iowa Test Method No. 212, and they continued to observe erratic strength behavior. Results of IDOT tests, performed on fly ash from Ottumwa generating station, are shown in Figure 22. Preliminary work at the MERL had also indicated a significant amount of variability in the compressive strength of Class C fly ash pastes. Thus a testing program was initiated to monitor the physical and chemical characteristics of these Class C fly ashes as a function of time.

Iowa fly ash production and sampling

The general locations of the power plants studied during this investigation are shown in Figure 23. Technical and operating details are summarized in Tables 8 and 9, respectively. In general, all of the power plants studied were of modern design and they all burned low-sulfur, sub-bituminous coal from the Powder River Basin near Gillette, Wyoming. Three of the power plants routinely added sodium carbonate to the raw coal feed to enhance the performance of their electrostatic precipitators.

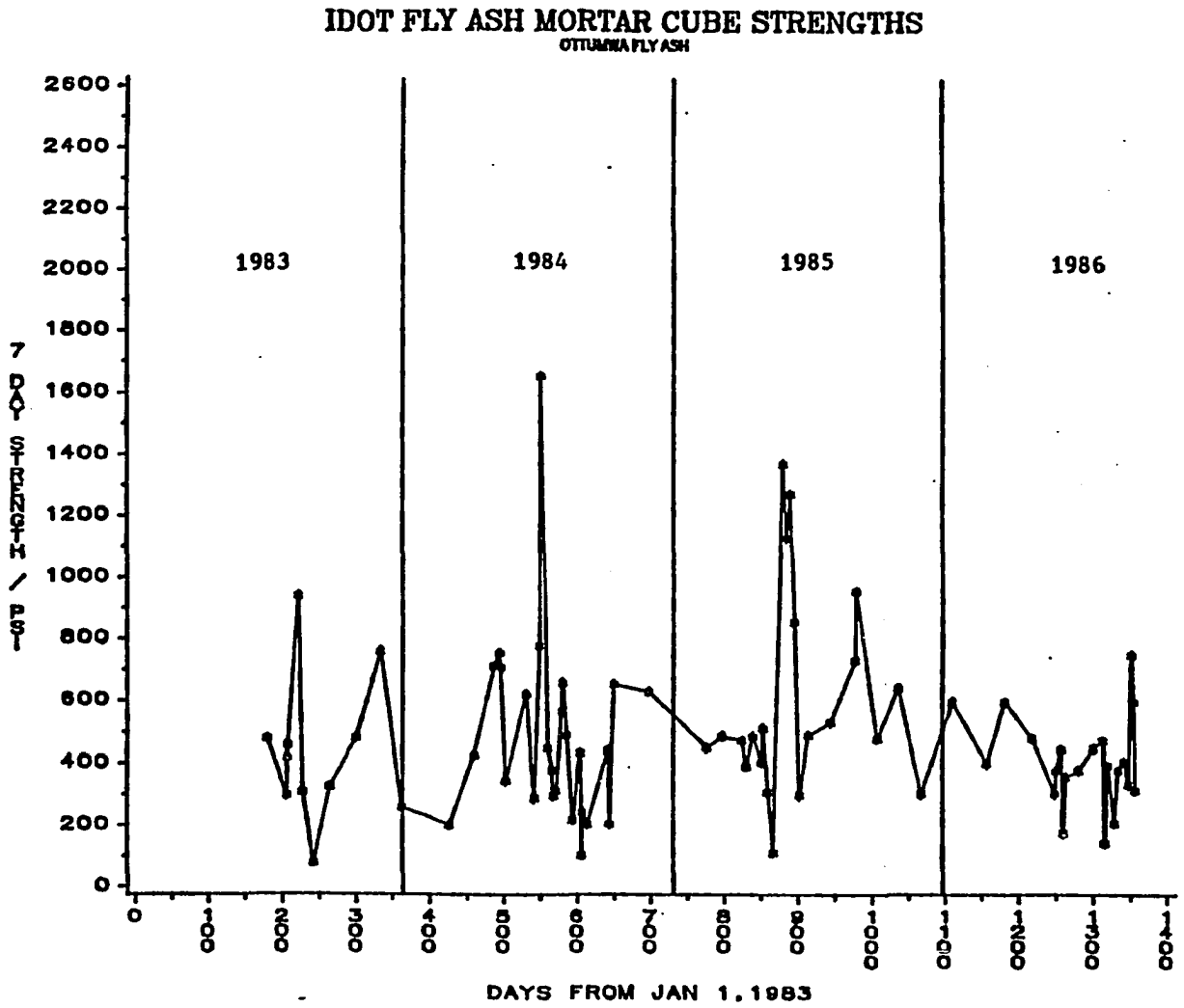


Figure 22. IDOT seven-day mortar cube strengths versus time for Ottumwa fly ash

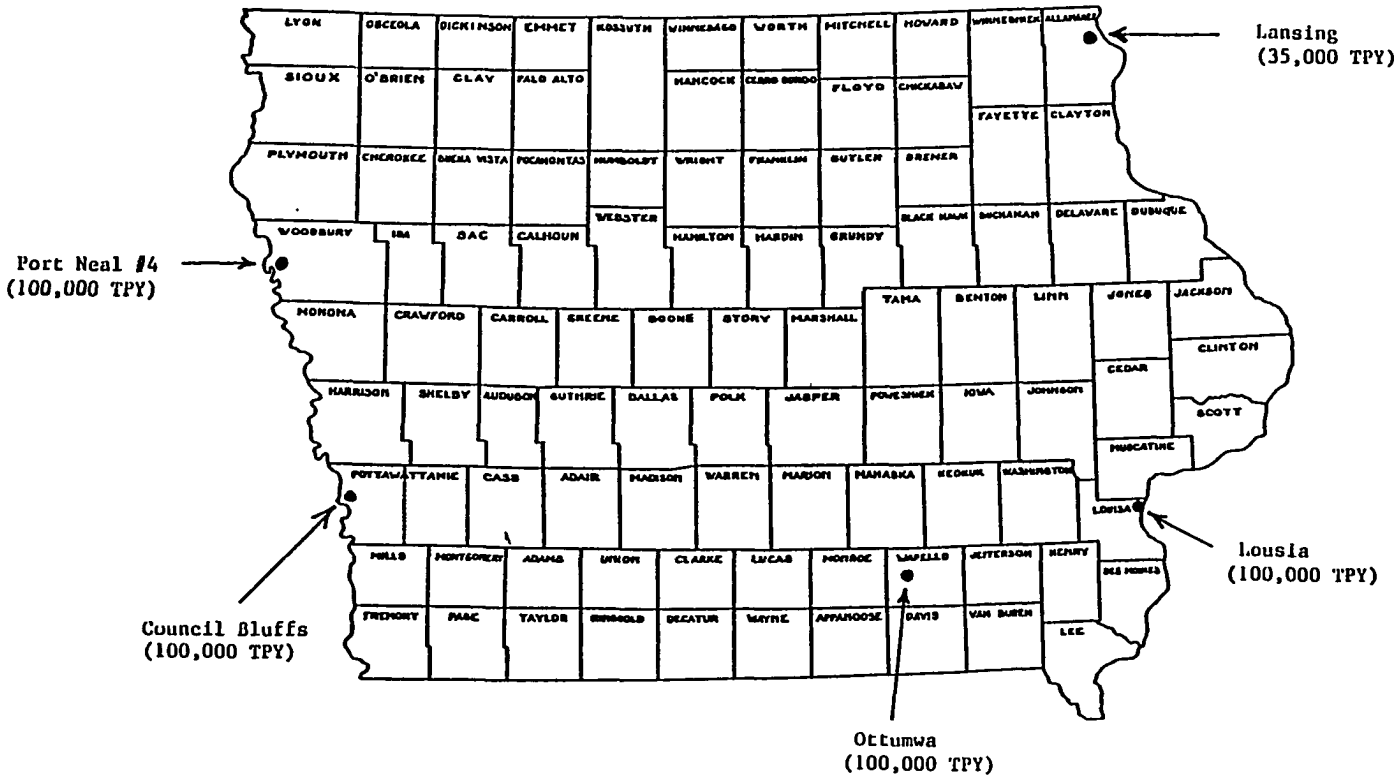


Figure 23. General locations and nominal fly ash production rates for the power plants studied in this research

Table 8. Technical details of the power plants investigated in this study

	Power Plant				
	Council Bluffs #3	**Lansing #4**	**Louisa**	**Ottumwa**	**Port Neal #4**
Boiler Type	Babcock-Wilcox	Riley Stoker	Babcock-Wilcox	Combustion Engineering	Foster Wheeler
Maximum Generating Capacity (net MW)	700	260	650	675	600
Year on Line	1978	1977	1983	1981	1979
Fly Ash Silo Storage Capacity (tons)	4000	300	3500	3500	5000
Precipitator type	Hot-ESP[a]	Hot-ESP	Hot-ESP	Hot-ESP	Currently being changed to cold ESP
Additive used to enhance ESP performance (lbs/ton of coal)	Sodium Carbonate (1 lb/ton)	NONE	NONE	Sodium Carbonate (1 - 3 lbs/ton)	Sodium Carbonate prior to 12/1/88 Future use uncertain

[a]Hot-ESP = hot side electrostatic precipitator.

Table 9. Operating details of the power plants investigated in this study

	Power Plant				
	Council Bluffs #3	**Lansing #4**	**Louisa**	**Ottumwa**	**Port Neal #4**
Coal Source (mine)	PRB,[a] Wyoming (Eagle Butte - Bell Ayr)	PRB, Wyoming (Eagle Butte - Bell Ayr)	PRB, Wyoming (Cordero)	PRB, Wyoming (Cordero)	PRB, Wyoming (Caballo)
Date when current coal contract expires	Dec. 31, 1997	Approx. 1996	Dec. 31, 2002	Approx. 2000	Dec. 31, 1998
Annual ash prod. (tons/yr)	100,000	25,000	72,000 (for 1986)	83,000 (for 1987)	100,200 (for 1987)
Approx. percent ash sold	16	40	100 (for 1986)	43 (for 1987)	36 (for 1987)
Typical Maintenance Cycle (tentative, 1988)	September for 4 weeks	Feb 28-Mar 5 May 29-Jun 11 Aug 28-Sep 3 Nov 27-Dec 10	Mid-Sept. thru Oct.	4/1 - 4/22/88 also 2 weeks in October	Early June
Start-up fuel	Fuel oil	Fuel oil	Natural gas or fuel oil	Fuel oil	Fuel oil

[a]PRB = Powder River Basin.

Fly ashes from Council Bluffs, Lansing, Ottumwa and Neal 4 power plants were selected to represent the range of Class C fly ashes available in Iowa. The sampling procedure that was used is described in ASTM C 618 and C 311 [5]. Briefly, grab samples were taken from each ash truck (approximately 20 tons) exiting the plant. After 20 grab samples were obtained, they were combined to form a sample representing 400 tons of fly ash. This sample was then tightly sealed in a clean one gallon paint can and mailed to the MERL.

Each sample, which represented a 400-ton lot of fly ash, was subjected to physical testing as per ASTM C 311 [5]. After five such samples were received, a chemical - physical test sample was made. The chemical - physical test sample was made by combining equal portions from each of the 400-ton lot samples, and hence, represented 2000 tons of fly ash. These chemical - physical test samples are referred to as "composite" samples by the ASTM.

After the first year of monitoring, it was observed that a power plant's operating conditions and maintenance cycle could significantly influence the chemical and physical properties of its fly ash. Hence, two very similar power plants (Ottumwa and Louisa) were chosen to study in detail. Grab samples of each fly ash were taken about three or four times per week for a duration of about four months. None of the samples were composited. Again, all of the samples were tightly sealed in metal paint cans and stored at the power plants until they were transported to the MERL. A total of about 100 samples were obtained from the two power plants.

Fly ash testing scheme

Two fly ash testing schemes were utilized in this study. The first utilized methods similar to those described in ASTM C 311 [5]. The second testing scheme was developed to monitor the self-cementing properties of fly ash pastes. A diagram of the overall (physical) testing scheme is shown in Figure 24. A diagram of the chemical testing scheme employed during this study is shown in Figure 25.

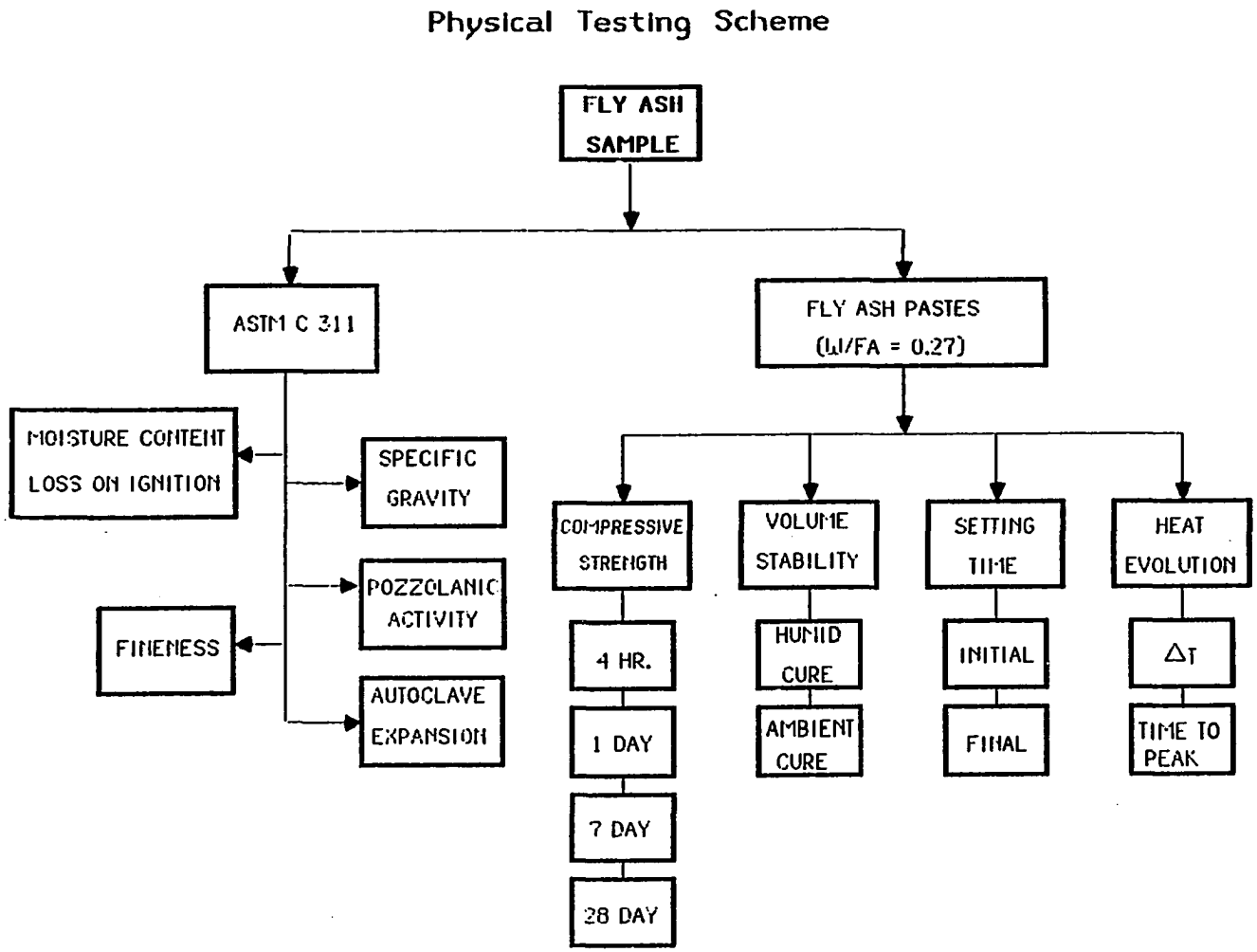


Figure 24. Diagram of the physical testing scheme employed during this research

OUTLINE OF EXPERIMENTAL PROCEDURES FOR
CHARACTERIZATION OF HIGH CALCIUM FLY ASH

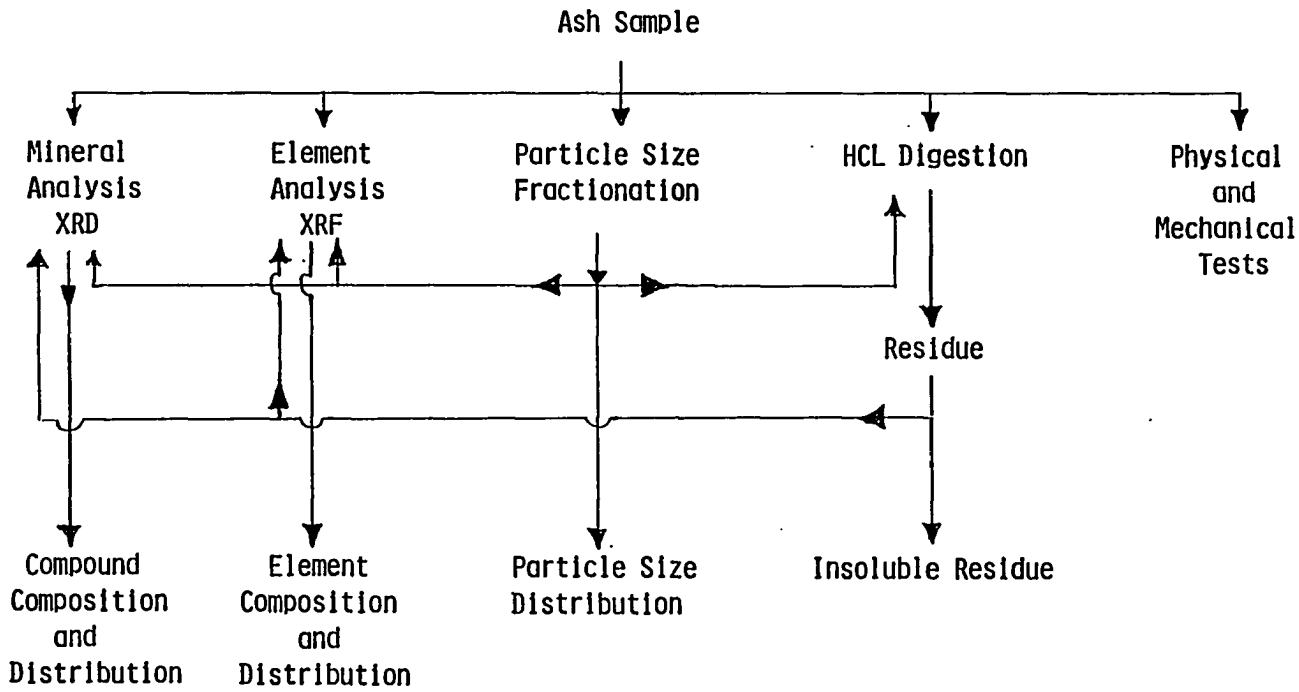


Figure 25. Diagram of the chemical testing scheme employed during this research

The two major differences between the testing methods used in this study and those listed in ASTM C 311 were: (1) a portland cement-fly ash mortar cube test was used in place of the lime-fly ash mortar test to assess the 7-day pozzolanic activity of the samples; (2) quick chemical methods (x-ray techniques) were used instead of the gravimetric and/or volumetric methods specified by the ASTM. The lime pozzolan test was replaced with a cement pozzolan test because researchers have found that the lime pozzolan test appears to be biased against Class C fly ashes [5, 88]. The change from classical (ASTM) chemical methods to x-ray methods was made to allow a throughput of a large number of samples. The analytical details of the chemical methods used in this study will be described later.

The fly ash paste testing scheme developed during this research program was used to study the compressive strength, volume stability, setting time and heat evolution characteristics of fly ash-water mixtures. The repeatability of the fly ash testing scheme was studied in detail. A multi-day, multi-operator test program indicated that the procedures, as defined below, were adequate for monitoring the physical characteristics of the fly ash pastes. The results obtained from the repeatability test program are summarized in Appendix A.

All of the fly ash paste mixes (except for the heat evolution test) were prepared at a water/fly ash ratio of 0.27. A typical batch consisted of 2000 grams of fly ash and 540 grams of deionized water. Hobart laboratory size mixers were used throughout the study. The paste mixing procedure consisted of: (1) adding water to the fly ash; (2) mixing at low speed for 30 seconds; (3) quickly scraping down the residue on the sides of the mixing bowl; (4) mixing at medium speed for 30 seconds. This mixing procedure produced a fluid paste that could be poured into the various molds for the different tests. A small vibration table was used to eliminate air voids from the fluid compressive strength and volume stability specimens.

The general details of the physical tests used during this project can be summarized as follows:

1. Compressive strengths were measured on one inch cube specimens tested after four hours, and 1, 7 and 28 days of moist curing. Three cubes were broken for each curing period. The cubes were loaded at a rate of 4000 pounds per minute until failure.

2. Volume stability characteristics were measured on 1 x 1 x 11 inch prisms (i.e., normal autoclave bar molds with gage studs positioned to maintain an effective gage length of 10 $\pm$ 0.1 inch). Two specimens were cast from each mix. One specimen was moist cured, the other was cured under ambient room conditions. Length measurements were taken periodically in accordance with ASTM C 490 [5].

3. Setting properties were evaluated using a soil pocket penetrometer. Test specimen container size was about 4.5 inches in diameter by 1 inch in depth. Penetrometer readings, in tons per square foot, were taken as a function of time. Most of the fly ash pastes set so quickly that readings needed to be taken at about 1 to 2 minute intervals for the first 15 minutes of the test. Hence, the specimens were not stored in a humid cabinet between readings. Initial set was defined as the first discontinuity in the pressure versus time curve. Final set was arbitrarily defined as 4.5 tons per square foot penetrometer bearing pressure.

4. Heat evolution of fly ash-water mixtures was monitored using a calorimeter constructed out of a DeWar flask and a thermocouple [63]. Test specimens consisted of 28.5 grams of fly ash and 10.0 grams of deionized water. The specimens were briefly mixed by hand before being inserted into the calorimeter. The calorimeter temperature was displayed directly on a chart recorder so a continuous record of temperature versus time was obtained.

Other Analytical Techniques

X-ray fluorescence spectrometry (XRF) was used to identify and quantify the major and minor elements present in the fly ash samples. A Siemens SRS 200 sequential x-ray spectrometer was used throughout this study. The spectrometer was fully computer controlled via an LC 200 interface and a PDP 11/03 microcomputer. The technical details of the quantitative routines that have been utilized at the MERL have been described elsewhere [83, 84]. Briefly, the XRF method is a comparative analytical technique. Samples of known elemental composition (i.e., standards) are used to calibrate the spectrometer for the elements of interest (Si, Al, Fe, Mg, Ca, K, Na, P, S, Sr, Ba and Ti in this instance). After calibration, specimens of unknown composition are analyzed and elemental concentrations are estimated. Existing software allows for the correction of interelement effects via multiple regression techniques and correction for x-ray tube drift [83]. The absolute

accuracy of the method was limited by the of lack of good standard reference materials for calibration. The relative error of the fly ash assays was estimated at about 3 to 5% of the amount reported for major elements (reported as oxides), and about 5 to 10% for minor elements. However, the overall precision of the method (i.e., repeatability of sample preparation and analysis) was very good. Typically, assays of duplicate samples were repeatable to 0.2% (absolute concentration) for major elements and 0.05% (absolute) for minor elements.

X-ray diffraction (XRD) was used to determine the crystalline compounds present in the various fly ash specimens. A Siemens D500 diffractometer was used throughout this study. The diffractometer was fully computer controlled via an LC 500 interface and a PDP 11/23 microcomputer. A copper x-ray tube was used for all analyses. Monochromatic radiation was obtained via a diffracted beam monochromater and electronic discrimination (pulse height analysis/discrimination). The diffractometer was equipped with medium resolution slits and it was operated in step scan mode.

Flame photometry was used to determine the concentration of specific elements (Na, K and Ca) in fluid samples. A Beckman DU-2 flame photometer was employed for this study. All analyses were conducted in an acidic matrix (HCl) using an oxygen-hydrogen flame. General details about the method can be found in Willard et al. [102], while details specific to this study can be found in ASTM C 114 [4].

Two additional methods were used during this study to enhance the characterization of Class C fly ashes. The first method employed was the Blaine fineness test. The second method consisted of separating fly ash into specific size fractions by sonic sifting.

The Blaine fineness test is most commonly used to measure the specific surface of portland cement [34]. The method has also been applied to fly ashes by making proper modifications [16]. Actually, the test was routinely used to estimate the specific surface of fly ash samples until 1973 [60]. The method was dropped from the routine testing program because a fly

ashes' carbon content significantly influenced the results of the test [16]. However, the residual carbon content (loss on ignition) of the Class C fly ashes investigated in this study was very low when compared to a typical Class F fly ash; hence, little bias is expected from the carbon content of the Class C fly ashes that were studied. The Blaine fineness tests were conducted in accordance to ASTM C 204 [4].

Several fly ashes were subjected to particle size separation by using an Allen-Bradley Sonic sifter (model L3P). The apparatus uses waves of sonic frequency to agitate particles on the sieves and thus, produces relatively quick and accurate size separation. Electroformed nickel metal sieves with nominal sizes of 45, 20 and 10 microns were used throughout this study. The fly ash particles passing through all of the sieves were also collected for subsequent analysis. Hence, four particle size fractions were obtained from the sonic sifter: 1) particles greater than 45 microns (denoted as >45); 2) particles smaller than 45 but larger than 20 microns (denoted as >20); 3) particles smaller than 20 but greater than 10 microns (denoted as >10); 4) particles smaller than 10 microns in diameter (denoted as <10).

RESULTS AND DISCUSSION

Results of ASTM Physical and Chemical Testing

The results of chemical and physical testing (also referred to as "total analysis") and of physical testing alone (also referred to as "routine analysis") of fly ashes obtained from Council Bluffs (CBF), Lansing (LAN), Ottumwa (OTT or OGS) and Neal 4 (NE4) power plants are summarized in Tables 10 and 11, respectively. Each table lists the mean, $\bar{X}$, standard deviation, S, the maximum observed value, MAX, the minimum observed value, MIN, and the number of samples, n.

Raw data are listed in Appendix B. Table I (Appendix B) lists the test results obtained from the ASTM chemical and physical testing of fly ash samples obtained between 1983 and 1987. Similar information concerning the physical test samples (routine tests) is summarized in Table II (Appendix B). The ASTM physical tests were conducted on samples that corresponded to 400 ton lots of fly ash. However, the majority of the chemical and physical tests were conducted on composite samples rather than grab samples. All of the composite specimens listed in Appendix B were prepared as specified in ASTM C 618 [5]. This fact must be emphasized because it may influence the interpretation of the information listed in Appendix B. The magnitude of the potential influence is currently unknown.

All of the fly ash samples tested during this phase of the study (189 total analysis samples and 685 physical test samples, taken over a 5 year interval) passed the specifications listed in ASTM C 618 [5]. In fact, few of the samples even approached the specification limits (see the MIN and MAX columns in Tables 10 and 11). Hence, one may assert that we are currently "overtesting" our fly ash sources. This may be so. However, it appears that the existing ASTM fly ash tests and sampling scheme may not adequately identify "bad" fly ash, especially if a power plant is approaching a maintenance shutdown. The major problem with the current ASTM methods is that they were created for Class F fly ashes. Class C ashes, which are enriched with alkaline earth elements (i.e., Ca, Mg, Sr and Ba), are drastically different from Class F ashes. The major items of

Table 10. Summary of results of chemical-physical testing of Iowa fly ashes from 1983 through 1987

Test	$\bar{X}$	S	MAX	MIN	$\bar{X}$	S	MAX	MIN	Specifications
Power plant	**Council Bluffs (n=37)**				**Lansing (n=31)**				**ASTM**
Moisture content, %	0.07	0.05	0.21	0.00	0.04	0.03	0.14	0.00	3.0 max.
Loss on ignition, %	0.40	0.14	0.76	0.19	0.45	0.19	0.77	0.20	6.0 max.
Fineness, % ret.	11.5	2.3	19.1	7.7	11.8	2.3	16.8	6.6	34 max.
7 day Pozzolan, %	89	5	99	80	89	4	99	80	
Autoclave Exp., %	0.11	0.03	0.15	0.05	0.10	0.03	0.17	0.04	0.8 max.
Specific Gravity	2.70	0.04	2.76	2.60	2.78	0.02	2.82	2.72	
28 day Pozzolan, %	93	8	117	81	90	7	103	75	75 min.
H_2O required, %	90	2	96	86	91	3	100	88	105
SiO_2, %	31.2	1.9	35.3	27.6	31.9	2.6	41.2	29.2	sum
Al_2O_3, %	16.6	0.7	18.0	15.1	16.2	0.7	17.6	14.7	≥ 50
Fe_2O_3, %	5.6	0.7	7.1	4.7	5.9	0.4	6.7	5.2	≤ 70
SO_3, %	3.25	0.50	4.37	2.22	3.91	0.51	4.88	2.84	5.0 max.
CaO, %	28.6	1.6	32.4	25.7	28.3	1.4	30.4	25.3	
MgO, %	5.92	0.61	6.82	4.90	5.97	0.55	7.30	5.13	
P_2O_5, %	1.04	0.29	1.71	0.60	0.92	0.18	1.33	0.61	
K_2O, %	0.28	0.06	0.38	0.18	0.29	0.08	0.54	0.2	
Na_2O, %	1.82	0.20	2.29	1.45	1.89	0.26	2.33	1.14	
Avail. Alkali, %	1.29	0.14	1.62	1.02	1.38	0.18	1.70	0.88	1.5 max.

Table 10. (Continued)

Test	$\bar{X}$	S	MAX	MIN	$\bar{X}$	S	MAX	MIN	Specifications
Power plant	**Neal 4 (n=46)**				**Ottumwa (n=75)**				**ASTM**
Moisture content, %	0.04	0.02	0.08	0.00	0.03	0.03	0.09	0.00	3.0 max.
Loss on ignition, %	0.29	0.07	0.50	0.16	0.27	0.06	0.42	0.17	6.0 max.
Fineness, % ret.	11.5	2.1	15.9	4.9	10.1	0.8	13.1	8.3	34 max.
7 day Pozzolan, %	91	6	104	79	92	5	104	76	
Autoclave Exp., %	0.07	0.02	0.11	0.02	0.05	0.03	0.10	-0.01	0.8 max.
Specific Gravity	2.61	0.08	2.74	2.42	2.64	0.04	2.72	2.54	
28 day Pozzolan, %	96	7	113	82	97	8	112	78	75 min.
H_2O required, %	90	2	100	86	89	2	96	83	105
SiO_2, %	34.5	2.3	41.1	30.0	33.0	2.2	38.2	29.5	sum
Al_2O_3, %	16.5	1.1	18.5	14.9	18.6	0.7	20.5	17.3	≥ 50
Fe_2O_3, %	5.8	0.4	6.3	4.5	5.5	0.4	6.4	4.8	≤ 70
SO_3, %	3.02	0.79	4.57	1.84	2.38	0.50	3.68	1.37	5.0 max.
CaO, %	25.4	1.6	28.1	22.1	24.9	1.0	27.3	22.3	
MgO, %	5.39	0.62	6.32	4.35	4.72	0.23	5.28	4.28	
P_2O_5, %	1.04	0.29	2.09	0.72	1.62	0.31	2.31	0.90	
K_2O, %	0.33	0.08	0.64	0.21	0.38	0.04	0.48	0.27	
Na_2O, %	2.25	0.24	2.84	1.78	2.27	0.46	3.28	1.33	
Avail. Alkali, %	1.44	0.31	1.84	0.80	1.55	0.37	2.62	0.84	1.5 max.

Table 11. Summary of results of physical testing of Iowa fly ashes from 1983 through 1987

Test	$\bar{X}$	S	MAX	MIN	$\bar{X}$	S	MAX	MIN	Specifications
Power plant		**Council Bluffs (n=112)**				**Neal 4 (n=153)**			**ASTM**
Moisture content, %	0.09	0.12	0.85	0.01	0.04	0.03	0.17	0.00	3.0 max.
Loss on ignition, %	0.43	0.20	1.58	0.17	0.30	0.06	0.47	0.16	6.0 max.
Fineness, % ret.	11.3	1.9	18.3	7.6	12.1	1.9	17.5	8.0	34 max.
7 day Pozzolan, %	89	6	103	74	91	6	107	77	
Autoclave Exp., %	0.10	0.03	0.15	0.03	0.07	0.02	0.13	0.02	0.8 max.
Specific Gravity	2.70	0.04	2.78	2.54	2.59	0.09	2.73	2.36	
Power plant		**Lansing (n=61)**				**Ottumwa (n=359)**			
Moisture content, %	0.04	0.03	0.15	0.00	0.03	0.02	0.13	0.00	3.0 max.
Loss on ignition, %	0.44	0.18	0.96	0.13	0.26	0.07	0.54	0.10	6.0 max.
Fineness, % ret.	11.1	2.1	15.8	7.1	10.1	1.1	14.5	7.4	34 max.
7 day Pozzolan, %	87	5	98	74	93	7	129	74	
Autoclave Exp., %	0.09	0.03	0.17	0.04	0.05	0.03	0.11	-0.01	0.8 max.
Specific Gravity	2.78	0.02	2.82	2.72	2.64	0.05	2.75	2.47	

the current ASTM fly ash tests of concern are: (1) the composite sampling scheme; (2) the available alkali test; (3) the pozzolanic activity test; and (4) the wet-sieved fineness test.

The composite sampling scheme was described earlier in this dissertation. A composite sample represents 2000 tons of fly ash and it consists of a linear combination of five physical test samples (each representing 400 tons). Test results of this study indicate that the compositing process tends to smooth out (or eliminate) extreme values. Both the physical test samples and the composite samples were subjected to the same basic tests (i.e., moisture content, loss on ignition, fineness, specific gravity, 7-day pozzolanic activity and soundness). Hence, by comparing the coefficient of variation and the range variation statistics for these tests, one observes that the compositing scheme tends to reduce the variation in the test results. This trend is illustrated in Figure 26. The line of equality depicted in these figures simply indicates the trend that one would expect if both series of tests produced exactly the same results. The mean values (average test results) were the same for both series of tests. This smoothing process is not totally bad because it also indicates that the composite samples should quickly produce a good estimate of the properties of an "average" fly ash sample. However, the penalty paid for this information is rather severe because the compositing step makes it difficult to predict the true variability present in a given source of fly ash. Hence, one must conclude that the chemical information presented in Table 10 is biased. The mean test results are reasonable but the standard deviation and range statistics are at best a lower bound to the true variation that exists in the various power plants.

The alkali content of fly ash from several of the power plants is of great concern. The concern stems from the potential problems that may occur when using the fly ashes in portland cement concrete. Cement alkalis normally have a potential to adversely influence the long term strength of concrete, cause physical disruption by reacting with alkali sensitive aggregates or to cause unsightly efflorescence on the surfaces of finished products [46,62]. Fly ash alkalis may (or may not) lead to similar problems; more research is definitely needed in this area. Whatever the

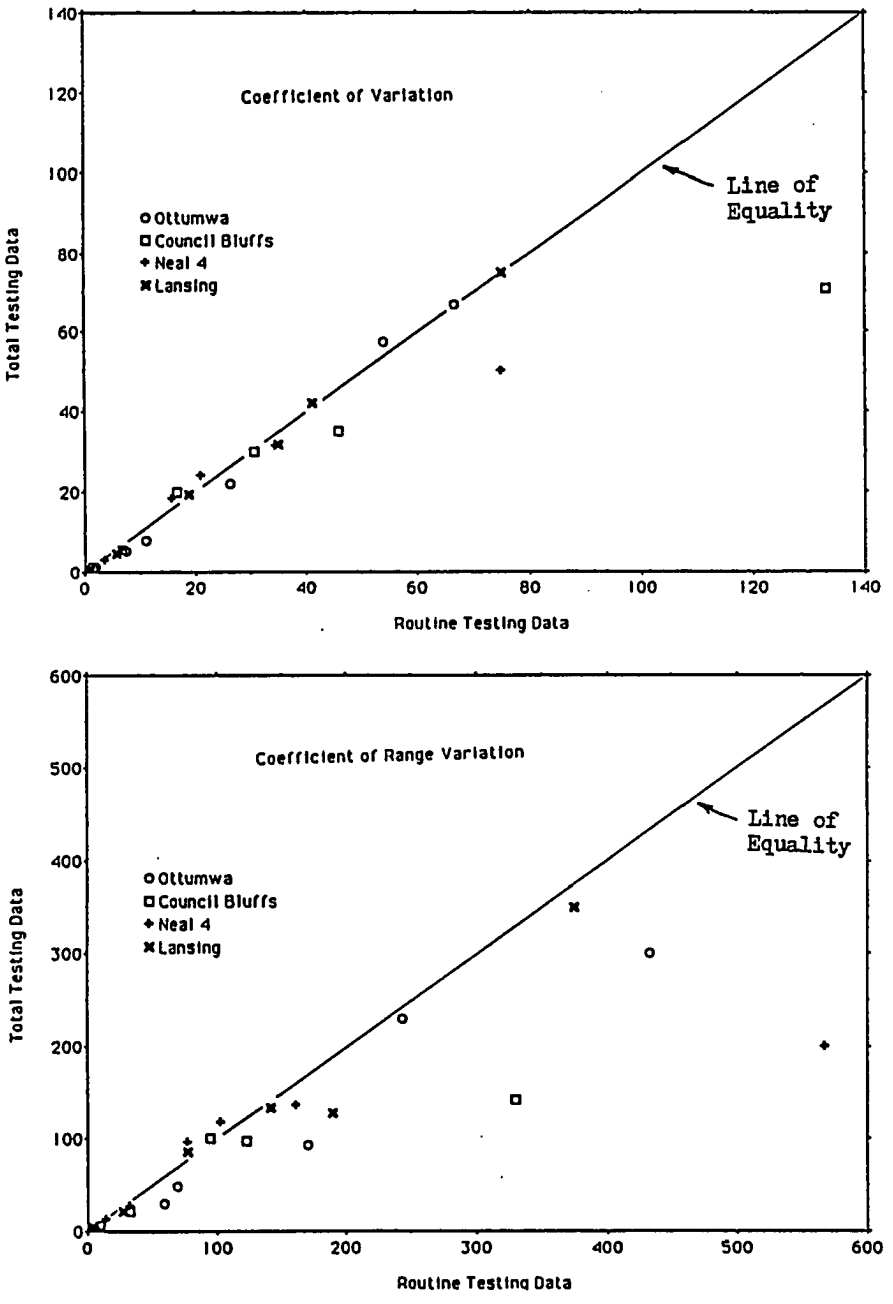


Figure 26. Diagram illustrating how the ASTM composite sample testing scheme reduces the variability of fly ash test statistics

case, a new test must be adopted for measuring the alkali content of Iowa fly ashes because the current test, the available alkali test, is not adequate [47].

The major problem with the available alkali test is that it underestimates the amount of alkali that may be leached into solution [47]. The test results (see Figure 27) clearly indicate that the 28-day curing period at 38 C, simply is not long enough to extract all the alkalis into solution. This same observation was made in 1956 by Brink and Halstead [12], although their work pertained only to Class F fly ashes.

The ASTM no longer requires a pozzolanic activity test for the physical test samples (i.e., 400 ton lots) of Class C fly ashes [5]. Only the chemical-physical test samples are subjected to a 28-day cement pozzolan test. Class F fly ashes are still required to be tested for pozzolanic activity (lime pozzolan test) on a sample by sample basis. The exact rationale behind this change is not clear; however, the results of this study (see Tables 10 and 11) indicate that the change should have little influence on the process of accepting or rejecting a given lot of fly ash. All of the Class C fly ashes tested at the MERL between 1983 and 1987, behaved satisfactorily in the cement pozzolanic activity test. However, as alluded to earlier, testing only composite samples (i.e., 2000 ton lots) may severely hinder the process of identifying if a power plant is producing subpar fly ash, especially if one must wait 28 days for test results.

The wet-sieved fineness test is another example of a test that may not be directly applicable to the analysis of Iowa Class C fly ashes. These Class C fly ashes contain a significant portion of water soluble compounds which may simply be washed into solution and through the sieve. Class C fly ashes contain very little residual carbon, so the test really only measures the coarse quartz particles in the fly ash. The determination of a particle size distribution curve would perhaps correlate better to the observed physical behavior of fly ash pastes or to the pozzolanic activity of fly ash-cement mortars and concretes.

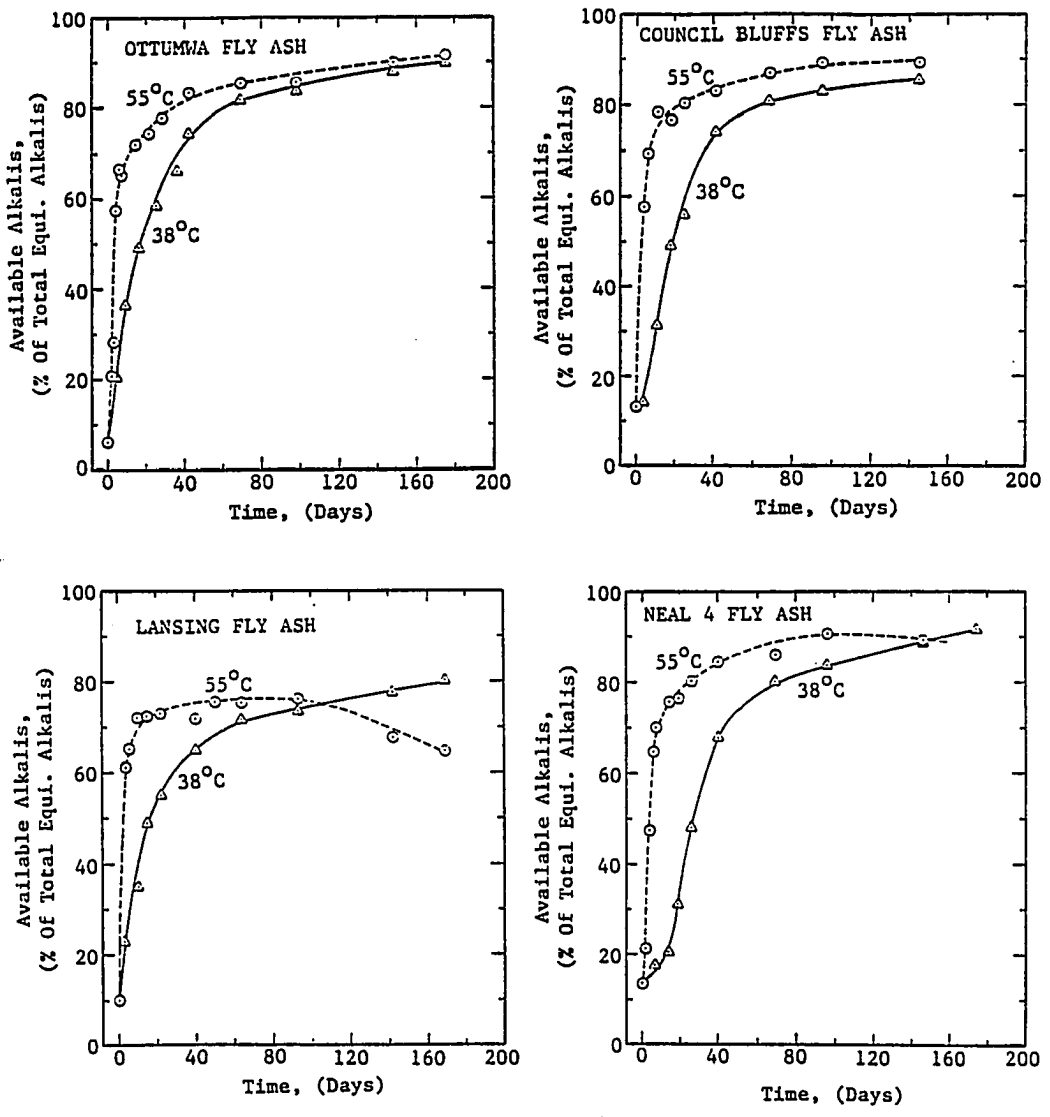


Figure 27. Effect of time and temperature on the mobilization of available alkalis in four Iowa Fly ashes

Results of Fly Ash Paste Testing

A statistical summary of the results obtained from the fly ash paste testing program is given in Table 12. Raw data and the results of correlation studies for the individual power plants are listed in Appendix C.

The results of compressive strength tests of 7-day old paste specimens made from Ottumwa, Council Bluffs and Lansing fly ashes are shown in Figures 28 and 29. These figures illustrate that the paste specimens had large variations in compressive strength as a function of sampling date. Paste specimens that were moist cured for other periods of time also exhibited nearly identical trends (see Appendix C). These test results are in agreement with those reported by IDOT personnel. Hence, one must conclude that the unexplained test variability reported by Isenberger [36] was due to changes in fly ash properties rather than simply poor test procedures. Also, both IDOT and MERL test results indicate how quickly the compressive strength of fly ash paste (or mortar) specimens can change as a function of sampling date. The variations in the compressive strength of pastes made from Ottumwa fly ash appear to be cyclical in nature (see Figure 28) and this trend will be discussed in detail later in this dissertation. Figure 30 illustrates the influence of moist curing time on the compressive strength of several fly ash paste specimens. Many of the fly ash samples studied during this research behaved similar to hydraulic cements.

Typical results obtained from the volume stability testing are shown in Figure 31. In general, most of the fly ash paste specimens expanded moderately during moist curing and shrank slightly during air curing. However, several of the fly ash samples from Ottumwa power plant exhibited severe expansive properties. In fact, such specimens generally fell apart (disintegrated) during the first few days of either moist or air curing. It was difficult to accurately measure the lengths of such specimens because their rates of expansion were very large immediately after removing them from the molds. More will be said about these samples later in this dissertation. The large majority of the specimens studied in this project did not exhibit problematic expansive behavior.

Table 12. A summary of fly ash paste statistics

Test	Council Bluffs (n=50)				Lansing (n=43)				Ottumwa (n=153)			
	$\bar{X}$	S	MAX	MIN	$\bar{X}$	S	MAX	MIN	$\bar{X}$	S	MAX	MIN
Compressive Strength (psi)												
4 hour	1127	365	2057	475	1955	524	2824	78	314	116	613	33
1 day	1483	454	2624	580	2847	1061	5074	316	549	366	2467	112
7 day	2409	1068	5669	820	4041	1443	6869	792	1084	887	4721	132
14 day[a]	3033	1355	5325	1011	4499	1622	8196	772	1198	995	5221	173
28 day	3769	1625	6681	921	5187	1729	8180	787	1393	1198	6038	148
56 day[a]	4807	996	6933	3402	6070	2113	9335	3024				
% Expansion @ 28 Days												
Air cured	-0.05	0.02	0.01	-0.10	-0.09	0.04	-0.02	-0.17	-0.04	0.03	0.12	-0.14
Humid cured	0.04	0.06	0.31	-0.03	0.04	0.04	0.16	-0.01	0.00	0.03	broke	-0.04
Setting time (minutes)												
Initial set	11	4	19	4	8	3	17	2	21	12	97	8
Final set	14	5	26	7	12	7	50	4	33	22	198	12
Heat Evolution												
Time to peak (min.)	29	11	58	13	26	9	56	15	40	14	82	11
ΔT (°C)	11	2.6	18	8	14	3.0	19	4	5	1.5	8	0.3

[a]n was less than the value denoted above for these tests, see the raw data.

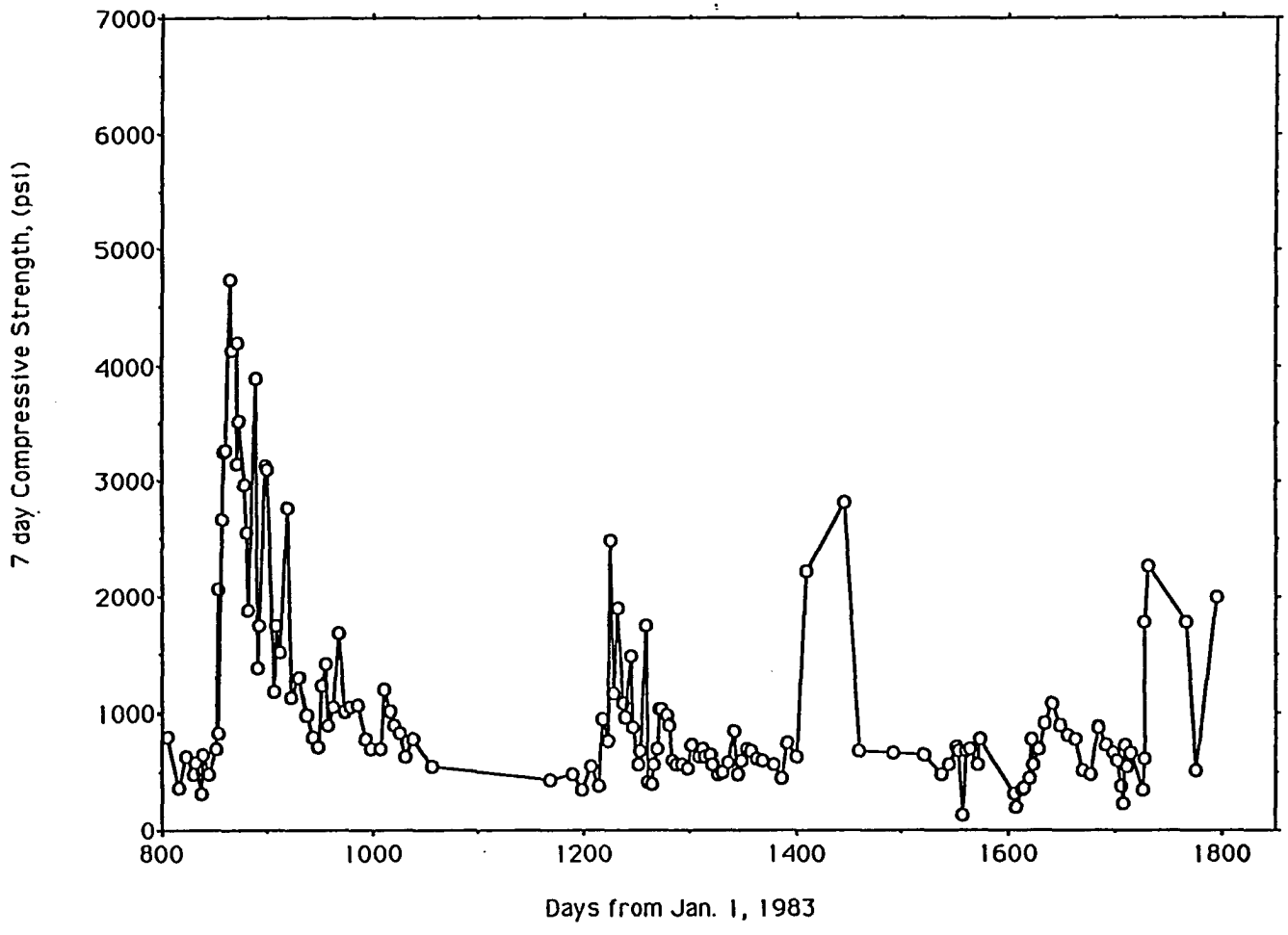


Figure 28. Seven-day compressive strength of Ottumwa fly ash pastes versus sampling date

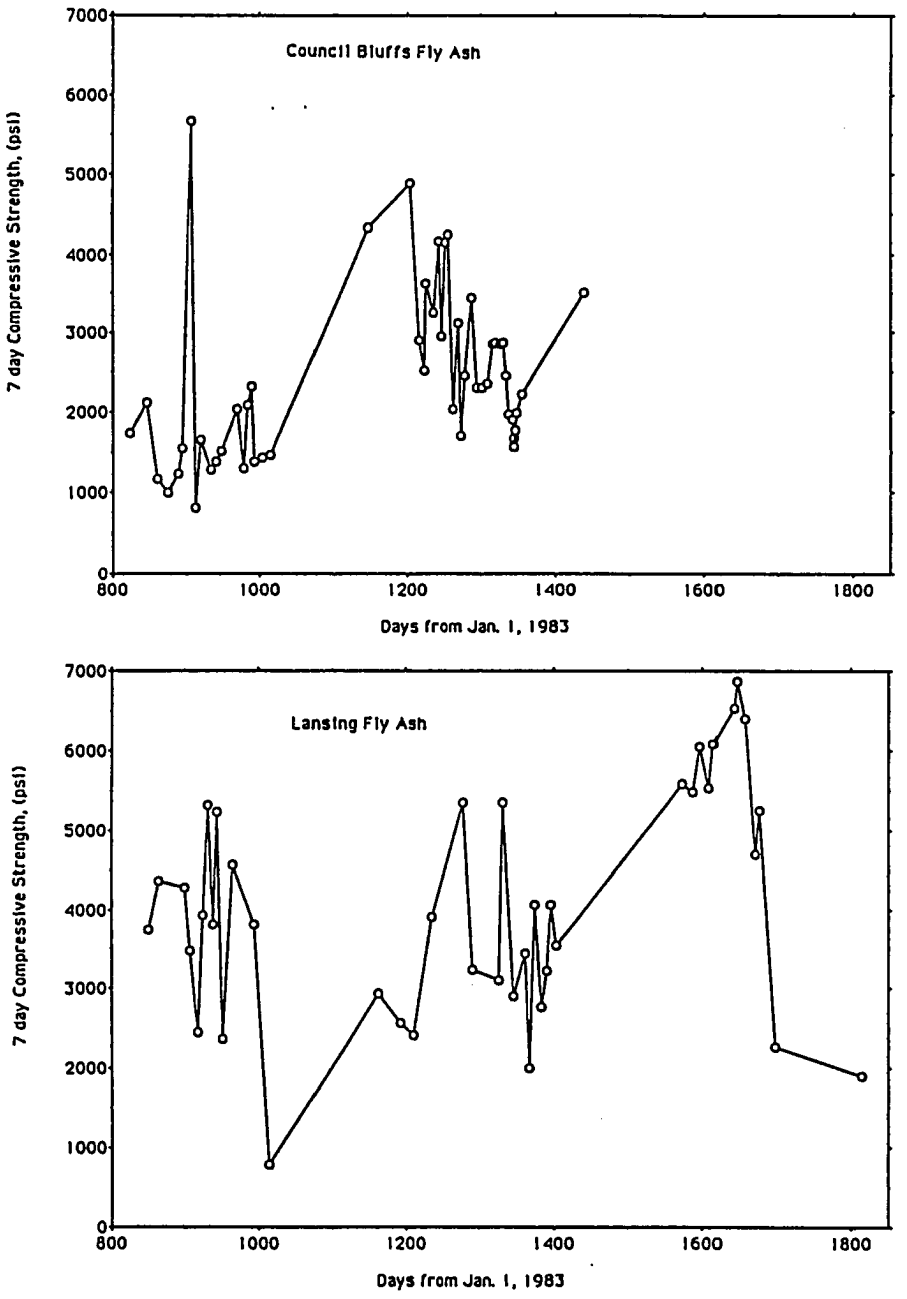


Figure 29. Seven-day compressive strength of Council Bluffs and Lansing fly ash pastes versus sampling date

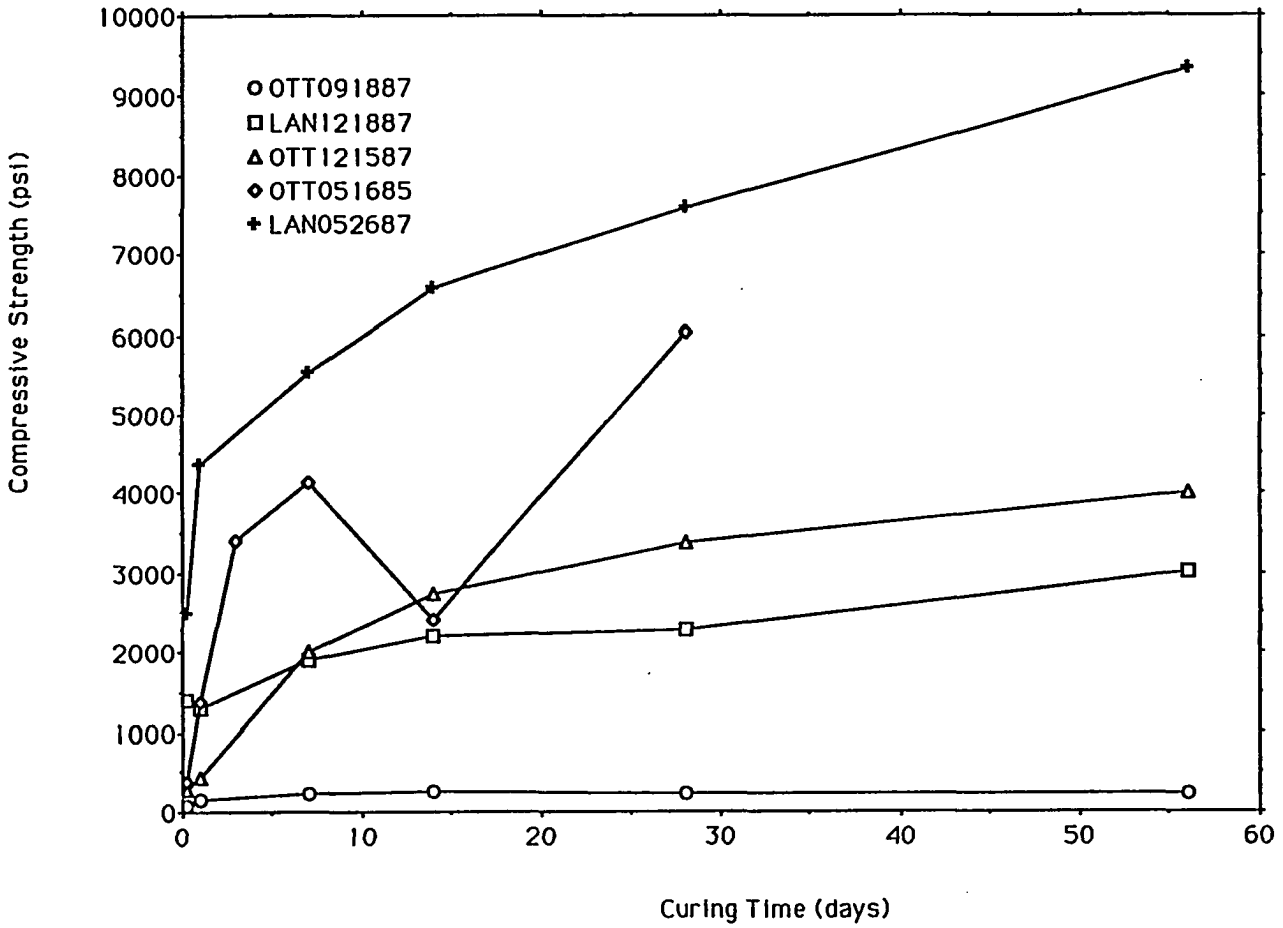


Figure 30. Compressive strength of fly ash paste specimens versus moist curing time

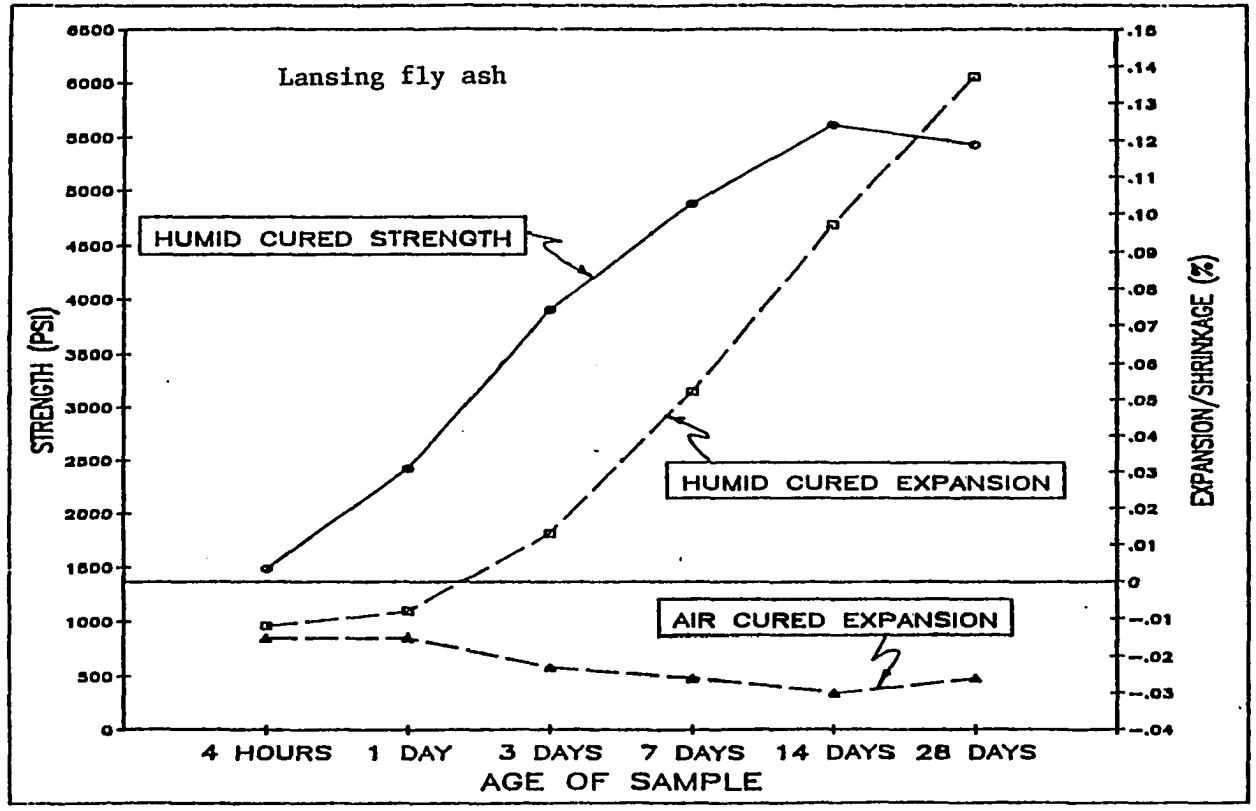


Figure 31. Volume stability of fly ash pastes for different types of curing versus time

Typical results obtained from the set time tests are shown in Figure 32. This figure illustrates the definitions of initial and final set that were used in this study. Generally, the fly ash paste specimens had initial set times of about 10 minutes and final set times of about 15 minutes. Hence, some field applications, such as soil stabilization or void filling, may be tricky unless proper retarders are found. Portland cement appears to be a reasonably good retarder for most of these fly ashes.

Typical results obtained from the heat evolution test are shown in Figure 33. Again, the various samples obtained from a single power plant exhibit drastically different behaviors as a function of sampling date. Fly ash from the Lansing power plant was generally the most reactive with water (i.e., highest ΔT), followed by the Council Bluffs fly ash and then the Ottumwa fly ash.

Correlation studies were performed in an attempt to define relationships between the various paste properties studied in this research project. The results of a correlation study utilizing all of the fly ash paste samples is summarized in Table 13. Abbreviations for the different variables were as follows:

4hour	=	4 hour compressive strength
1day	=	1 day compressive strength
7day	=	7 day compressive strength
14day	=	14 day compressive strength
28day	=	28 day compressive strength
56day	=	56 day compressive strength
%expair	=	air cured expansion @ 28 days
%exphu	=	humid cured expansion @ 28 days
initial set	=	initial set time
final set	=	final set time
peakT	=	peak temperature
finaltime	=	time required to reach peak temperature
deltaT	=	temperature rise (peak temp. - initial temp.)

Linear correlation coefficients (Pearson) were generated using a standard statistical package. Please note that all correlation matrices are symmetric about their main diagonals.

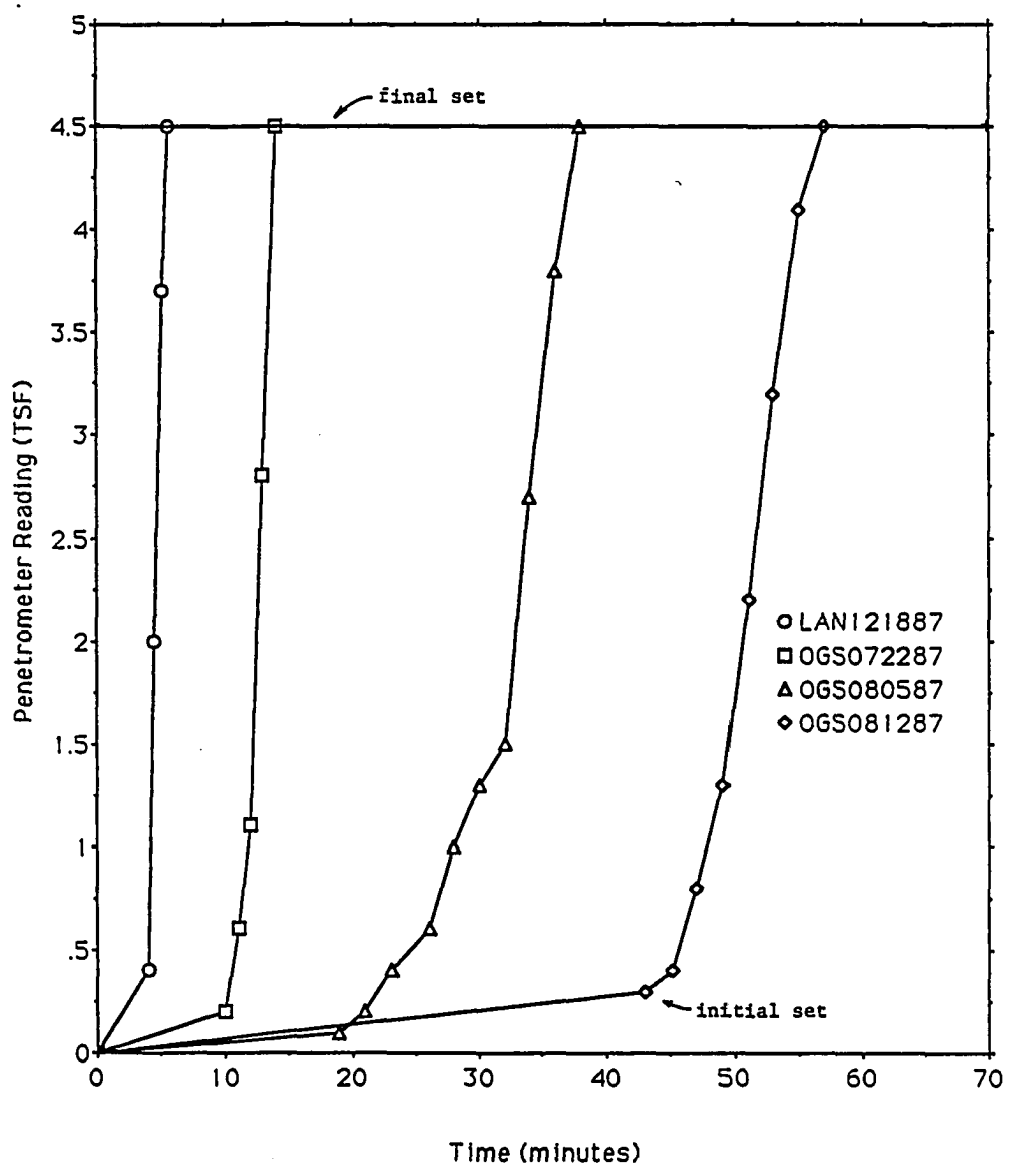


Figure 32. Results of time of set testing for several Iowa fly ash pastes

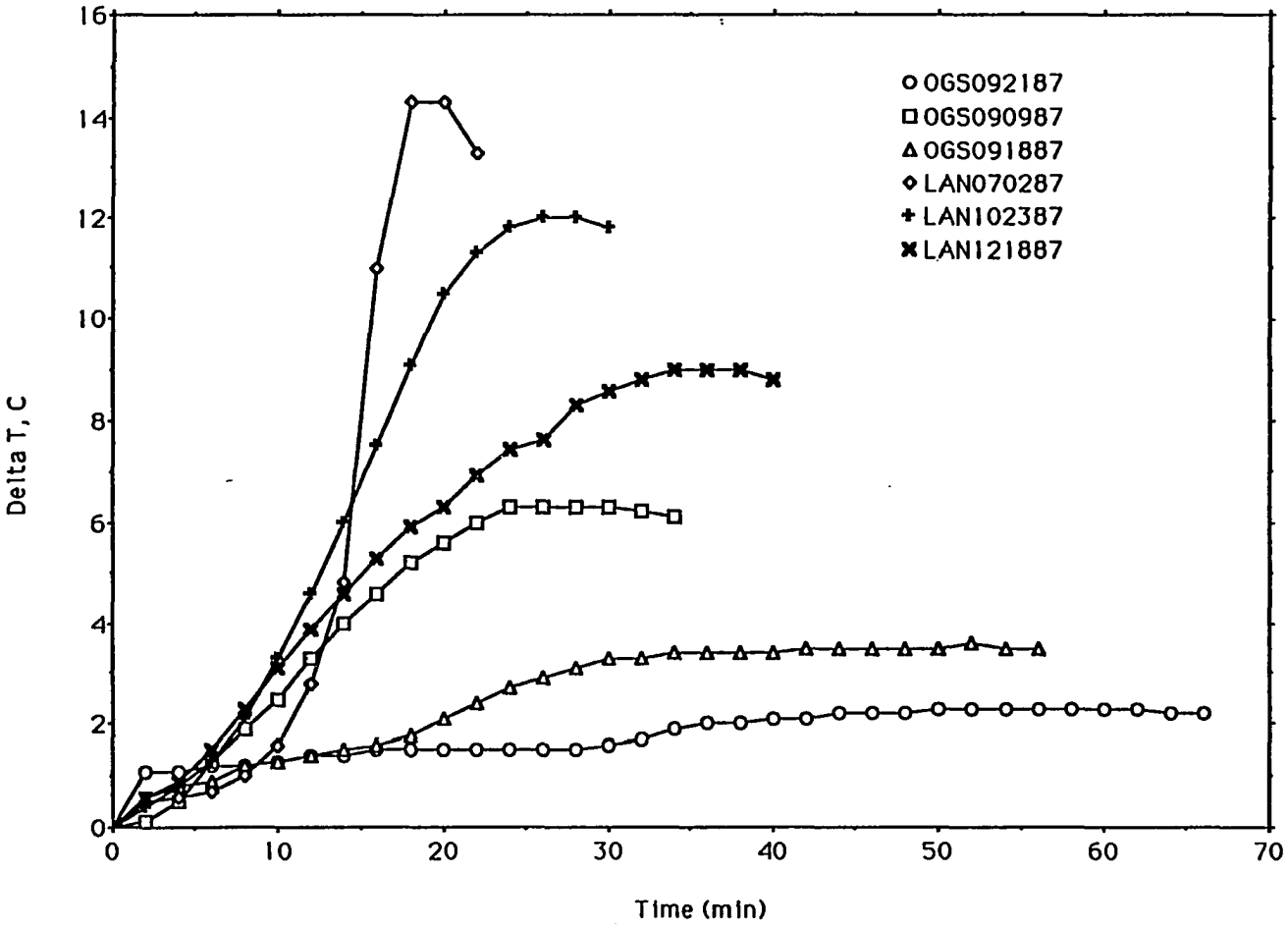


Figure 33. Results of heat evolution testing for several Iowa fly ash pastes

Table 13. Correlation matrix for the Iowa fly ash paste specimens (all pastes, 1985-1987, N = 264 in most instances)

	4 hour	1 day	7 day	14 day	28 day	56 day	% expair	%exphu.	initial set	finalset	finaltime	delta T	peak T
4 hour	1												
1 day	.93488	1											
7 day	.8593	.92195	1										
14 day	.87476	.90988	.96427	1									
28 day	.89163	.90487	.9404	.96211	1								
56 day	.8671	.88144	.92057	.9377	.95378	1							
% expair	-.56632	-.63594	-.76088	-.71764	-.63376	-.69047	1						
% exphumid	.40919	.3326	.37545	.45311	.41501	.44215	-.19975	1					
initial set	-.21635	-.18874	-.20323	-.22044	-.18253	-.1728	.20649	-.22027	1				
final set	-.31895	-.27807	-.30257	-.33083	-.30961	-.29927	.27353	-.24704	.95627	1			
finaltime	-.19694	-.29304	-.3437	-.32489	-.31149	-.29959	.11188	-.22788	-.13702	-.15501	1		
delta T	.88228	.74598	.63358	.70828	.74968	.7015	-.31081	.47807	-.23173	-.3478	-.17603	1	
peakT	.80687	.64878	.56338	.65332	.6923	.62569	-.24239	.492	-.25843	-.37785	-.12783	.96324	1

Strong correlations were observed between several of the paste variables studied in this project. The most obvious correlations were between compressive strengths measured at different curing times, between compressive strength and temperature rise, between initial set and final set, and between final set and the time required to reach the peak temperature.

Several of the more interesting trends are shown in Figures 34, 35 and 36. Figure 34 illustrates the relationship between the 7-day and 28-day compressive strengths of the fly ash pastes. Linear regression of the data yielded the equation listed in the figure. Figure 35 illustrates the relationship between initial set and final set. Again, linear regression of the data yielded the equation listed in Figure 35. Figure 36 illustrates the relationship between the four-hour compressive strength and the temperature rise (line estimated using linear regression).

Results of Additional Chemical Tests

The purpose of this phase of the research project was to conduct a detailed investigation of the chemical constitution of the Iowa high-calcium fly ashes. The samples used in this analysis were all physical test samples, each representing 400 tons of fly ash. This was done to avoid the "smoothing" problems (as described earlier) that appear to be associated with the composite samples.

Bulk mineralogy

Typical diffractograms of raw (as-received) fly ashes from the various power plants are shown in Figure 37. All of the fly ashes exhibited very similar bulk mineralogies. Each of the fly ashes contained alpha-quartz, lime, periclase, anhydrite, tricalcium aluminate (or an altered mineral similar in structure and reactivity to tricalcium aluminate) and often a mineral similar to tetracalcium trialuminate sulfate. Also, a diffraction peak was often found at about 3.9 Å in many of the fly ash samples; this peak has not yet been assigned to a specific compound (it may be a weak reflection from anhydrite or possibly sodium sulfate). Each of the fly ashes exhibited a glass halo that reached

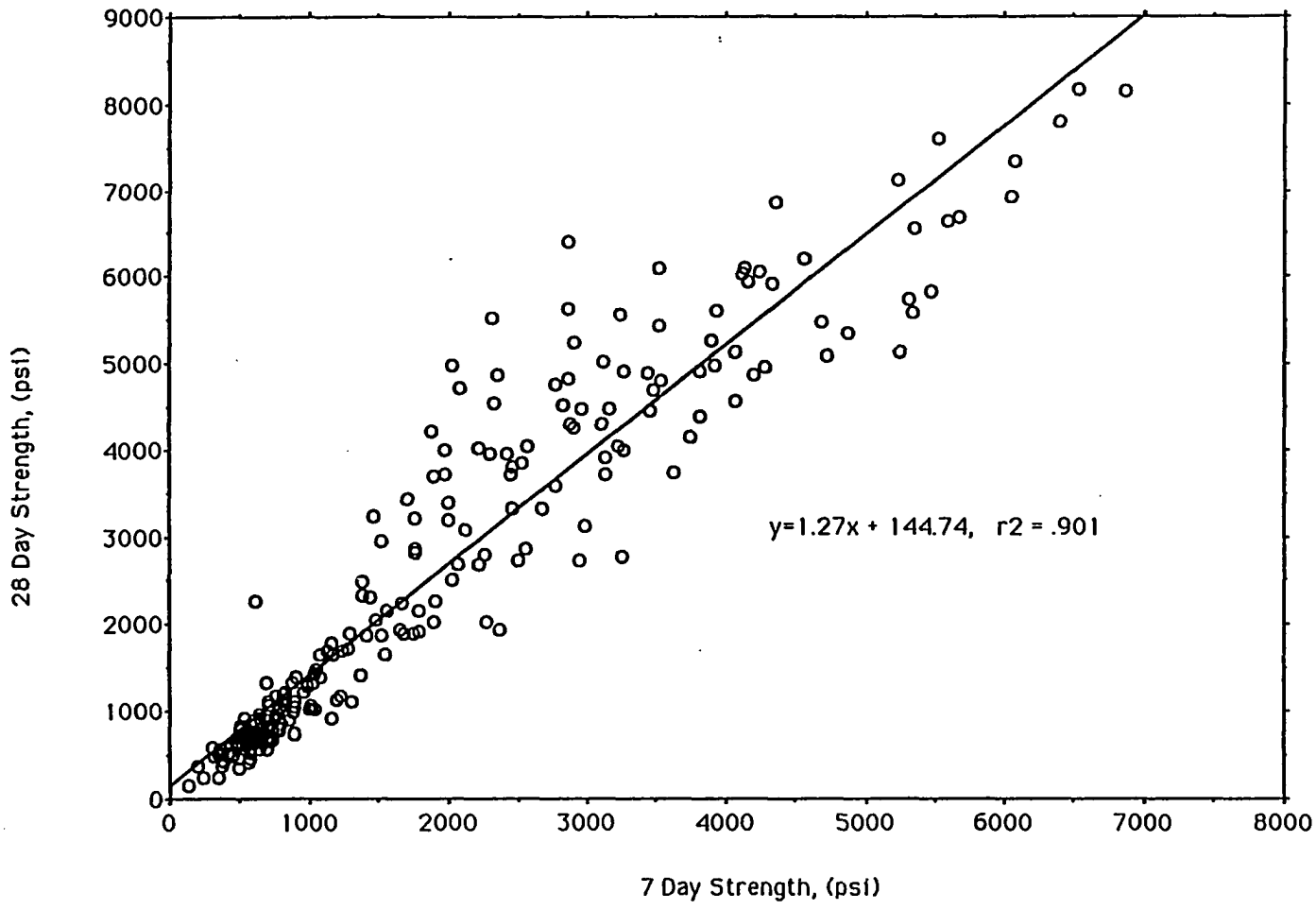


Figure 34. Relationship between seven-day and twenty-eight-day compressive strengths of Iowa fly ash pastes

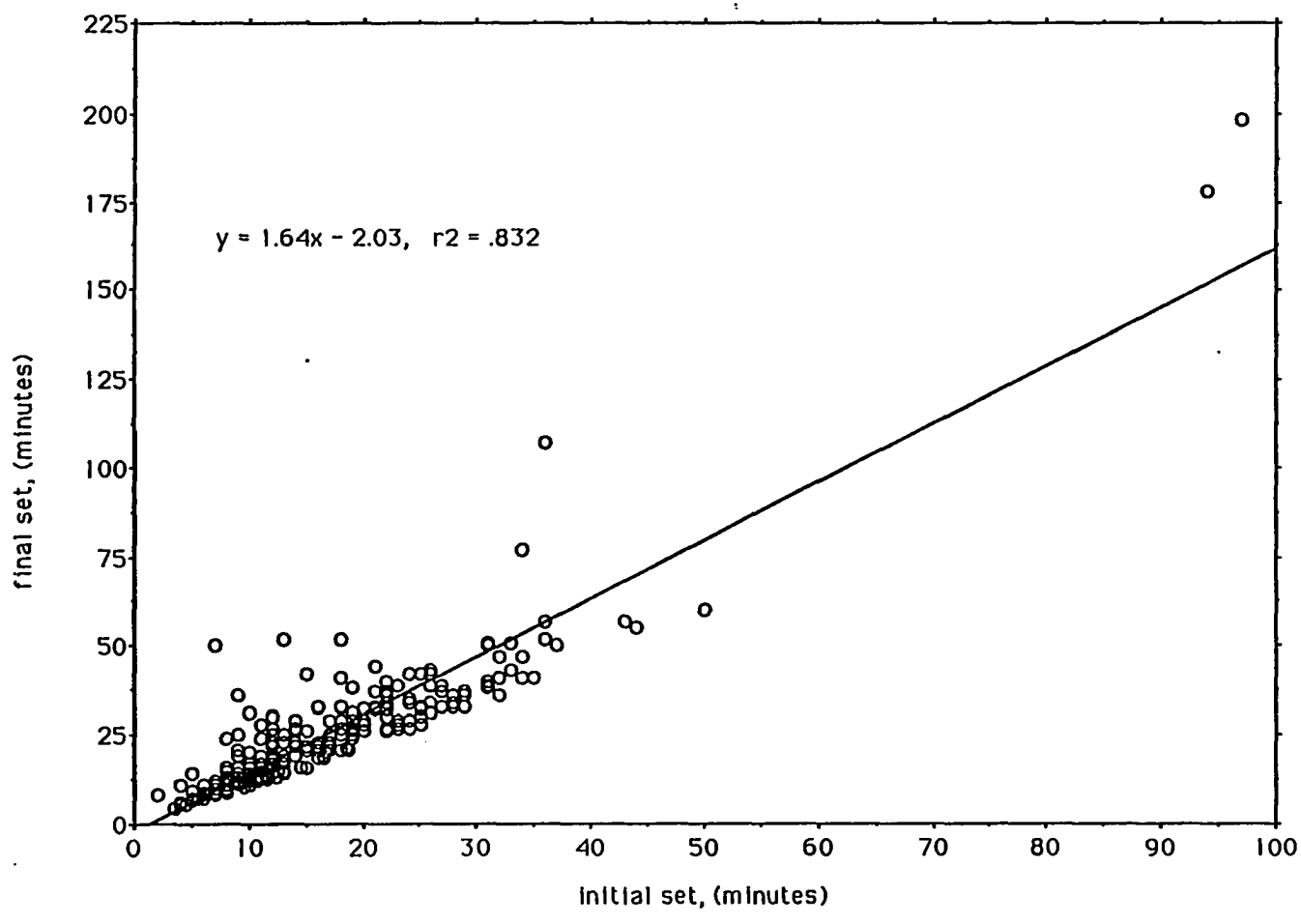


Figure 35. Relationship between initial set and final set for Iowa fly ash pastes

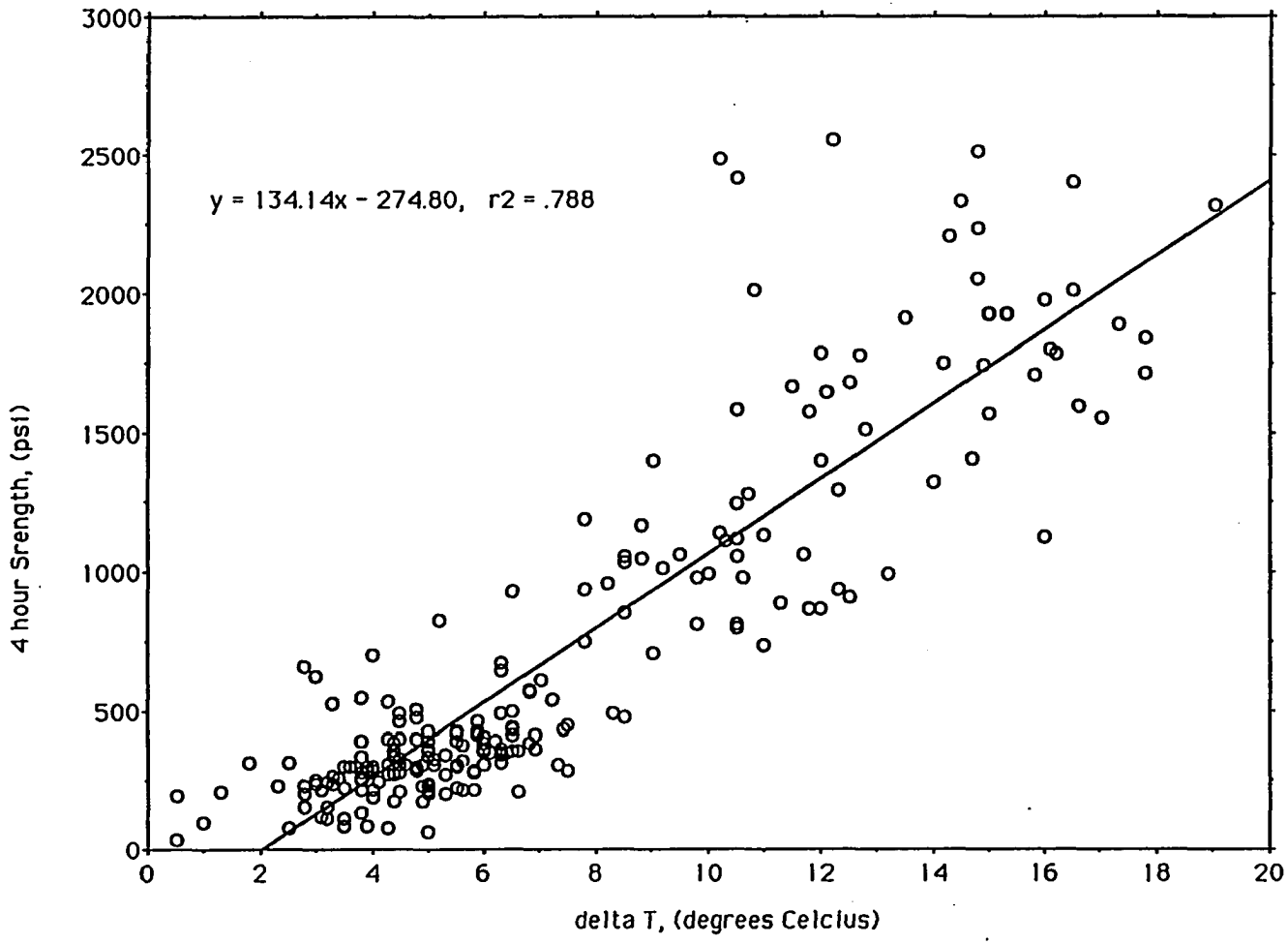


Figure 36. Relationship between four-hour compressive strength and temperature rise for Iowa fly ash pastes

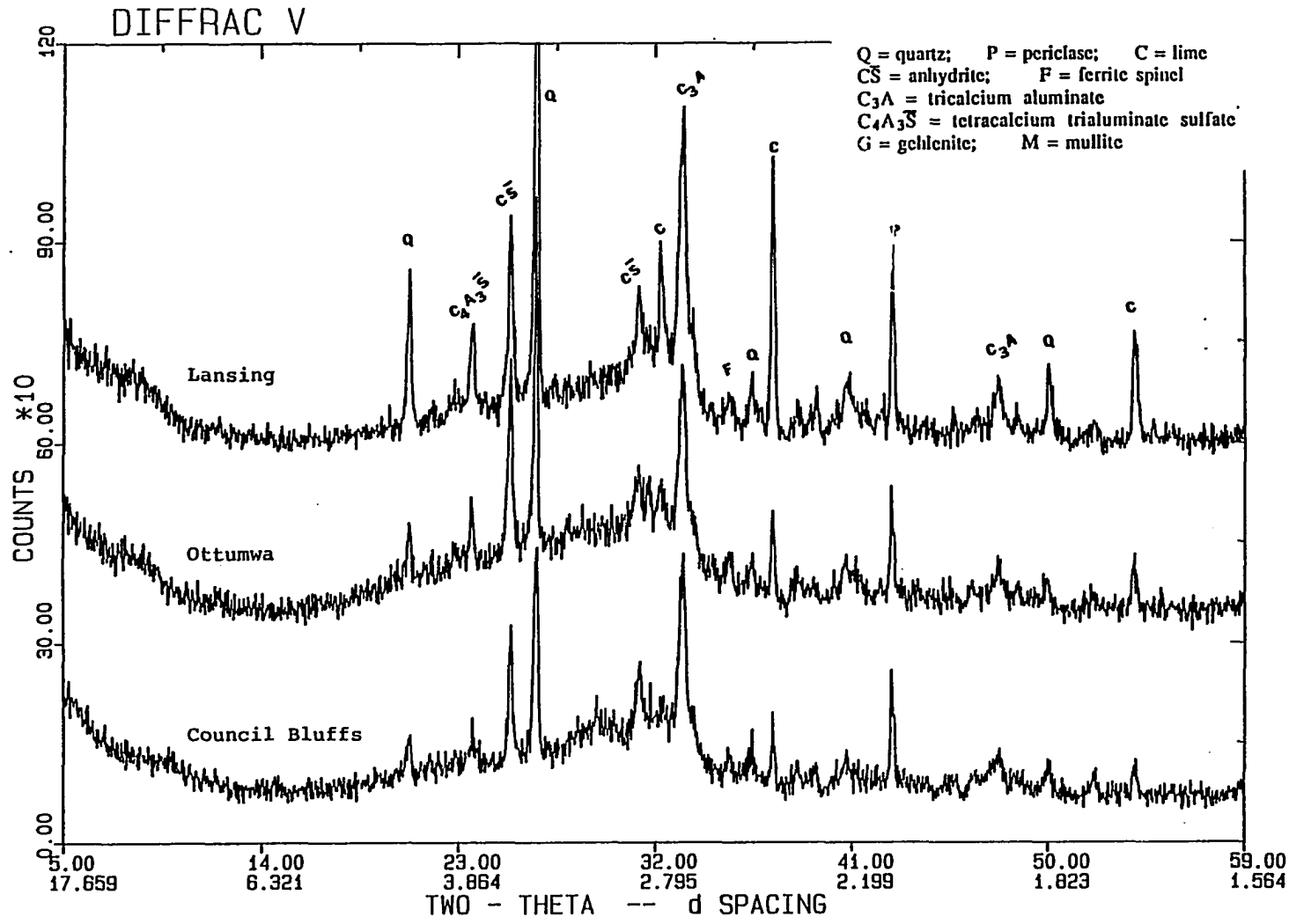


Figure 37. *X-ray diffractogram of "typical" fly ashes from three different Iowa power plants*

a maximum intensity above 30 degrees 2-theta (Cu K-alpha radiation). This scattering halo has been attributed to the presence of a calcium aluminate or a calcium aluminum silicate glass by other researchers [21, 58].

Table 14 summarizes the results of elemental analysis of the three fly ashes. In general, all of the fly ashes contained more than about 25% CaO. All of the fly ashes met the general requirements specified by the ASTM for Class C fly ash.

To obtain additional information about the glass phases and minor components present in the various fly ash samples, the raw fly ashes were digested in hot acid (6N HCl) as described in ASTM C 114 [4]. Flame photometry was used to estimate the concentrations of Na, K and Ca in the acid soluble fraction of the fly ash. The portion of the fly ash that was insoluble in the hot acid was washed, dried and then subjected to x-ray analysis.

Figure 38 shows the results of XRD analysis of the acid insoluble residue obtained from the three fly ashes. The acid digestion process removed many of the minerals present in the raw fly ashes, it also appeared to remove the majority of the glass that exhibited a scattering halo above 30 degrees 2-theta (i.e., the calcium aluminate or calcium aluminum silicate glass). In fact, all three of the fly ashes were quite soluble in hot HCl (about 70% soluble, see Table 14). The major minerals identified in the XRD patterns were alpha-quartz, mullite and magnetite (? some hematite). At least one minor mineral remains unidentified in the diffractograms.

The glass remaining after the HCl digestion appears to be high in silica. It is apparent that the glass scattering halos (see Figure 38) have shifted back to about 23 degrees 2-theta (Cu K-alpha radiation) for all of the fly ashes. This scattering halo is more indicative of a Class F (low-calcium) fly ash [21, 58]. Hence, we appear to have made Class F fly ashes out of the three Class C fly ashes by a simple acid extraction. The chemical assays of the acid insoluble fraction of the various samples support this interpretation (see the lower half of Table 14).

Table 14. Bulk compositions of three Iowa fly ashes before and after acid extraction (wt. %)

Oxide	Council Bluffs	Lansing	Ottumwa
	Before Acid Extraction		
SiO_2	30.6	31.1	34.6
Al_2O_3	15.3	15.9	18.5
Fe_2O_3	5.5	5.3	4.8
Sum	51.4	52.3	57.9
SO_3	3.3	3.9	3.7
CaO	28.8	28.1	24.9
MgO	5.8	5.7	5.3
Na_2O	2.0	1.6	3.3
K_2O	0.3	0.3	0.4
P_2O_5	0.9	1.2	1.1
TiO_2	1.0	1.4	1.4
SrO	0.4	0.4	0.5
BaO	0.7	0.6	0.8
%Acid Soluble	74	67	73
	After Acid Extraction		
SiO_2	71.9	67.7	64.2
Al_2O_3	8.4	10.9	12.6
Fe_2O_3	5.3	6.9	7.0
Sum	85.6	85.5	83.8
SO_3	1.0	1.0	0.8
CaO	4.8	4.7	4.9
MgO	1.6	1.4	1.3
Na_2O	1.4	0.7	1.7
K_2O	0.5	0.4	0.5
P_2O_5	0.1	0.2	0.2
SrO	0.2	0.2	0.2
BaO	2.3	2.2	1.8

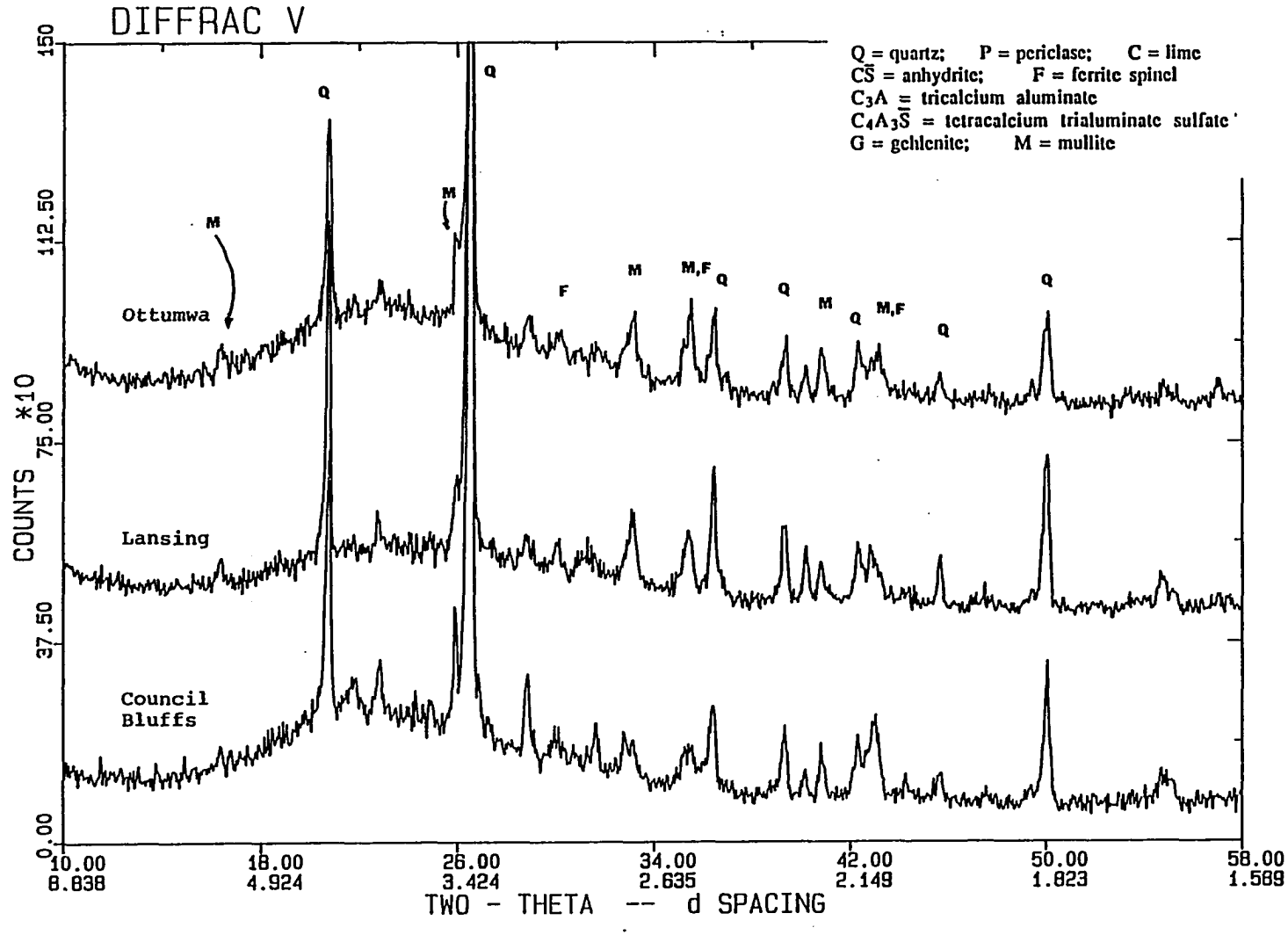


Figure 38. X-ray diffractogram of the acid insoluble fraction of three Iowa fly ashes

Flame photometry analysis of the acid soluble portion of the fly ashes indicated that about 60% of the Na, 50% of the K and 50% of the Ca had been extracted from a given bulk fly ash. These numbers should be regarded as only semi-quantitative at this time because of possible (unexpected) interferences in the flame photometry method.

Particle size separation (via a sonic sifter) was also very helpful in enhancing the identification of the minor crystalline compounds and different glasses present in the bulk fly ashes. Diffractograms of the various particle size fractions of fly ashes from Lansing and Council Bluffs power plants are shown in Figures 39 and 40, respectively. The fly ash from Ottumwa power plant exhibited particle size-mineralogy trends similar to the other two and, for brevity, will not be presented here.

The fly ash obtained from Lansing power plant showed minor changes in mineralogy when comparing the coarse fraction (>45 microns) to the smaller size fractions. One apparent trend indicated that alpha-quartz tended to accumulate in the larger (i.e., >45 and >20 micron) particle size fractions. The remaining two size fractions investigated (i.e., the >10 and <10 micron fractions) appeared to become enriched in lime, periclase and both calcium aluminates and sulfates. The glass scattering halos in these two size fractions appeared different from the halos observed in the larger size fractions. All of these observations are, of course, only qualitative.

The fly ash obtained from the Council Bluffs power plant shows a rather distinct mineralogy for each of the different size fractions. Again, the mineralogy of the coarse size fractions (i.e., >45 and >20 micron sizes) tended to be dominated by silicate type minerals such as alpha-quartz, a mineral similar to gehlenite (a melilite structural group) and a small amount of mullite. The major peaks for tricalcium aluminate, anhydrite, periclase and lime were still evident in the diffractograms but were of low intensity. The glass scattering halos for the >45 and >20 micron size fractions were distinctly different from two smaller size fractions. The mineralogy of the smallest size fraction (<10 micron particles) was especially interesting because it did not contain alpha-quartz. The major

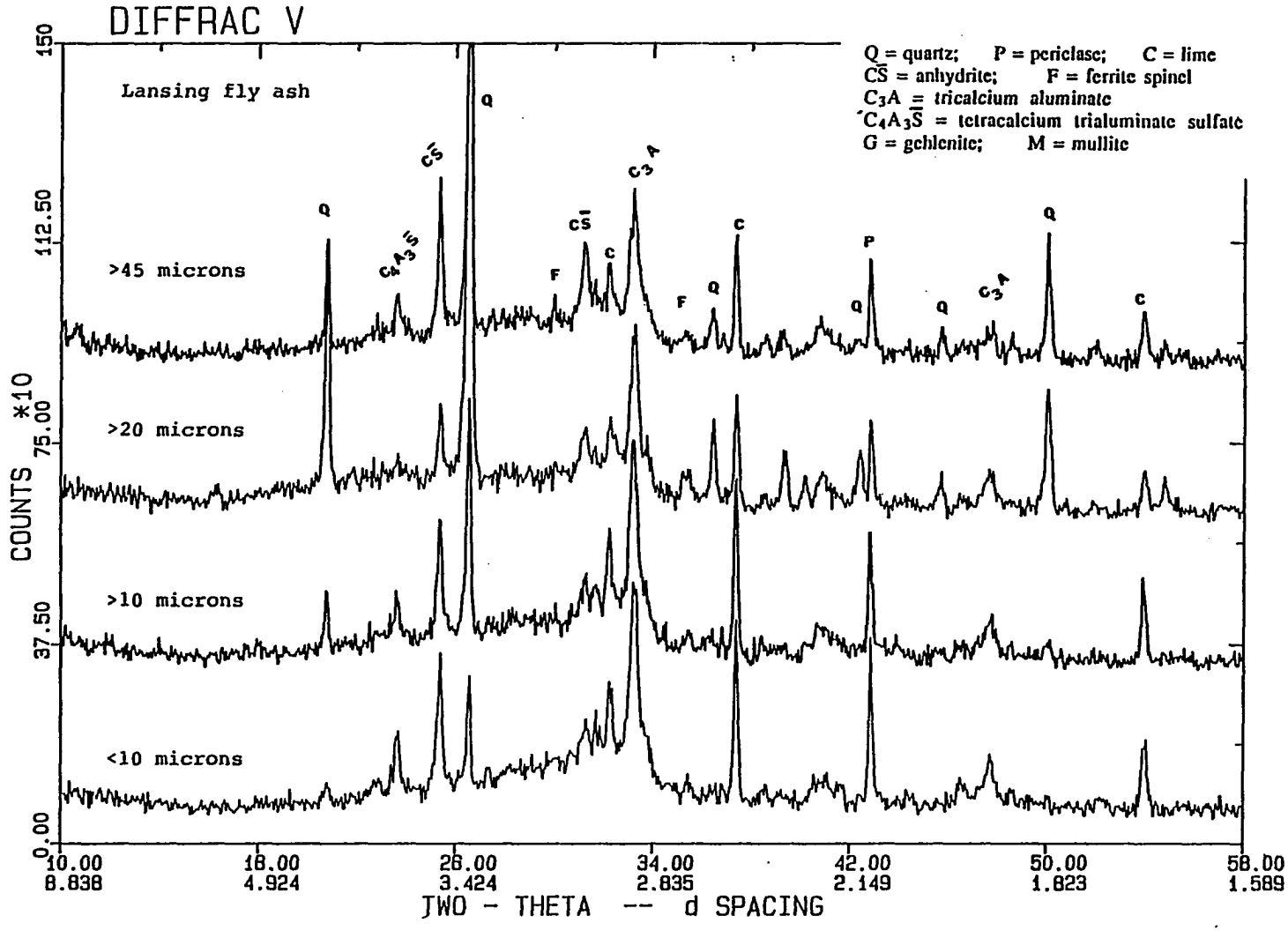


Figure 39. X-ray diffractogram of four different particle size fractions of Lansing fly ash

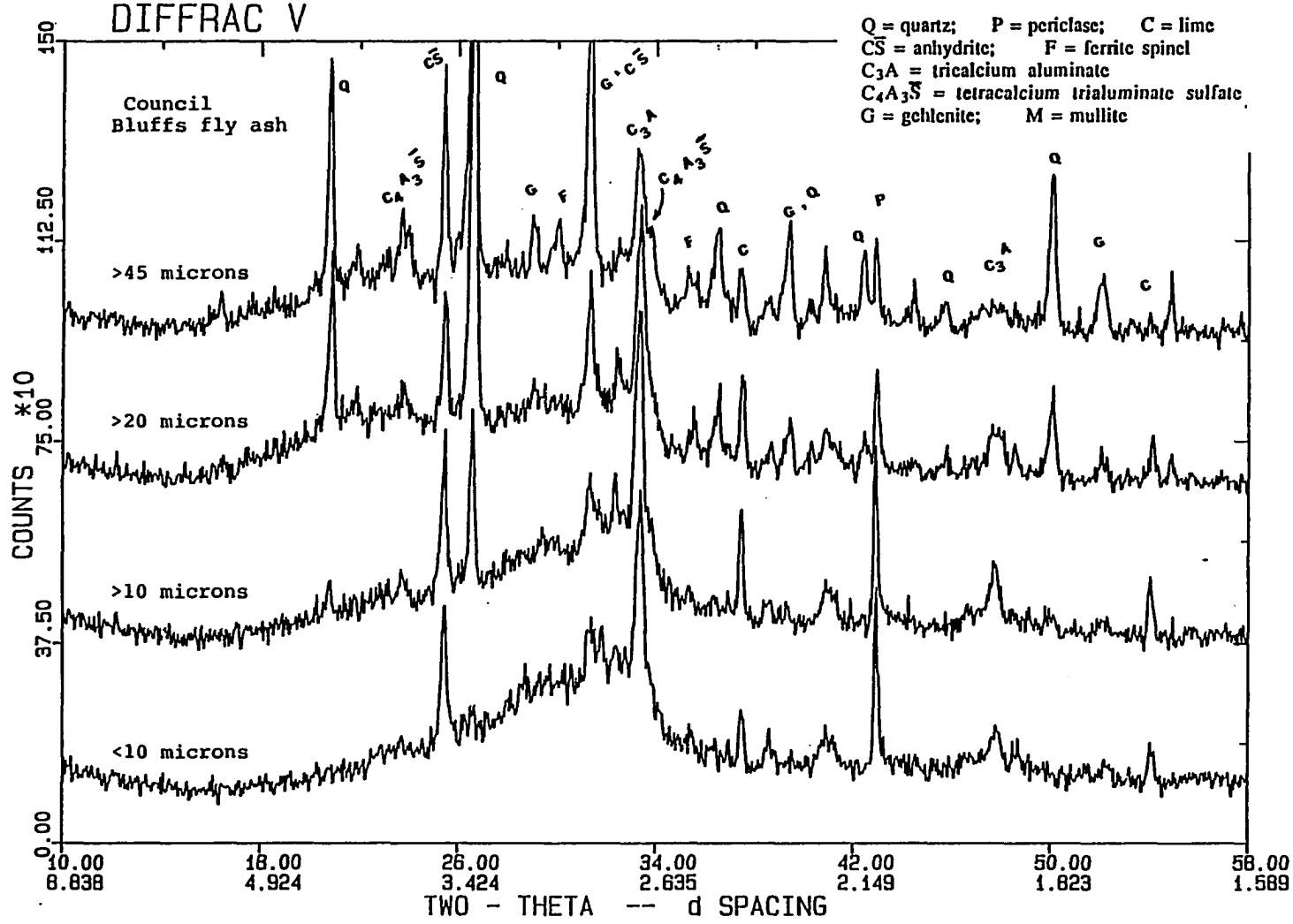


Figure 40. X-ray diffractogram of four different particle size fractions of Council Bluffs #3 fly ash

minerals in the <10 micron size fraction were anhydrite, lime, periclase and a mineral similar to tricalcium aluminate.

Table 15 summarizes the results of chemical analysis of the various particle size fractions of the Council Bluffs and Ottumwa fly ashes. The analyses were performed using XRF and a loose powder technique described elsewhere [84]. The values are semi-quantitative at this time, because both mineralogical and particle size effects were ignored during the analyses; however, interelement corrections were performed. Trends were quite distinct and did tend to agree with both the XRD results and with results reported by other researchers [31]. One must acknowledge that Hemmings and Berry [31] did not find distinct differences in mineralogy between the various size fractions of the fly ash that they studied, but they did find a relationship between mineralogy and density fraction. This discrepancy could be due to the rather low concentration of calcium present in their fly ash (i.e., 10% CaO versus >25% for this study). In general, the chemical analyses of the various particle size fractions of the Council Bluffs and Ottumwa fly ashes (see Table 15) indicated that the alkaline earth elements (Mg, Ca, Sr, Ba), and their commonly associated anion group, sulfur trioxide, tended to accumulate in the smaller size fractions at the expense of Si.

The glass halo present in the <10 micron particle size fraction, strongly indicated the presence of a calcium aluminum silicate glass (as previously hypothesized by Diamond [21] and Mehta [58]). Since no silicate bearing minerals were identified in the diffractogram of the <10 micron size fraction, we must conclude that the glass contains about 20% SiO_2.

Quantitative estimates were made of several of the crystalline compounds present in fly ash from Ottumwa power plant. Ottumwa fly ash was chosen for analysis because it had the largest variability in physical (paste) properties of all the power plants studied. Hence, these analyses were conducted in an attempt to help explain the high variability in physical properties of the pastes.

Quantitative x-ray diffraction (QXRD) analysis of fly ash is a complex problem due to: (1) small amounts of the compounds present; (2) numerous compounds in the ash with overlapping peaks;

Table 15. Bulk chemical composition of various particle size fractions of two Iowa fly ashes (wt. %)

Oxide	Decreasing particle size (values in microns) >45	>20	>10	<10
	Council Bluffs Fly Ash			
SiO_2	39	41	25	21
Al_2O_3	16	14	17	19
Fe_2O_3	5.3	5.3	5.9	6.4
Sum	60	60	48	46
SO_3	3.2	2.6	3.7	4.2
CaO	22	25	32	36
MgO	5.2	5.5	7.5	8.5
Na_2O	2.0	2.8	2.7	2.5
K_2O	0.4	0.3	0.2	0.2
P_2O_5	0.8	0.8	1.2	1.4
BaO	0.5	0.5	0.7	0.9
SrO	0.4	0.4	0.5	0.5
	Ottumwa Fly Ash			
SiO_2	35	35	28	24
Al_2O_3	20	18	19	19
Fe_2O_3	5.4	4.8	4.9	5.0
Sum	60	58	52	48
SO_3	2.2	2.2	2.8	3.2
CaO	18	22	26	29
MgO	4.2	4.6	5.1	5.8
Na_2O	2.7	2.8	2.8	2.7
K_2O	0.5	0.4	0.4	0.3
BaO	0.8	0.8	0.9	1.0
SrO	0.5	0.5	0.6	0.6

(3) the presence of the glassy phase; and (4) isomorphous substitution. As of this writing, quantitative evaluation of the amounts of compounds present are, at best, estimates only; nevertheless, it is necessary to define the cause(s) of the paste variations and to provide input to a rational characterization method. Table 16 summarizes the results of QXRD on 10 raw Ottumwa fly ash samples, which traversed the low to high strength paste region shown on Figure 28. The values shown on Table 16 are expressed relative to the concentrations of the various compounds present in the OTT051685 sample. Note, that the variation in the relative amount of tricalcium aluminate present in a given sample roughly corresponds to variation in the compressive strength of the paste specimens (see Figure 41). Obviously the cause of the variation in paste properties is more complicated than simply tricalcium aluminate content. However, the Ottumwa fly ash does not normally contain much free lime or tetracalcium trialuminate sulfate, so one may speculate that a tricalcium aluminate-anhydrite reaction should control the early setting and hardening relationships in these fly ash pastes. If this is so, then the early pore solution chemistry may be dominating the formation of hydration products, and hence, the physical properties of Ottumwa fly ash pastes.

Fly ash hydration products

Iowa high-calcium fly ashes are very reactive with water; this fact has been emphasized in both an earlier report [72] and also in the paste section of this dissertation. The major hydration reactions appear to occur between tricalcium aluminate and the sulfate bearing compounds (anhydrite and tetracalcium trialuminate sulfate) present in a fly ash. Also, one must consider the minor components such as lime, periclase (although this compound may be hard-burnt) and the alkalis (sodium and potassium) present in the fly ash. Obviously, composition and microstructure of the hydration products will influence the physical properties of the fly ash pastes.

The diffractograms shown in Figure 42 clearly indicate that the major hydration products in the various paste specimens were ettringite, monsulfoaluminate and straetlingite, and this is in good agreement with previous researchers [8, 45, 48, 64]. However, there is no simple

Table 16. Results of QXRD on a series of samples of fly ash from Ottumwa generating station

Sample	Day from 1/1/83	Concentrations (wt%) relative to OTT051686				
		SiO_2	$CaSO_4$	CaO	MgO	C_3A[a]
OTT011385	743	1.23	1.10	0.71	0.75	0.49
OTT022085	781	0.58	1.19	1.39	0.95	0.57
OTT031585	804	0.75	1.13	1.40	0.81	0.71
OTT032685	815	0.79	1.01	0.82	0.79	0.58
OTT050785	857	0.85	1.05	1.17	0.83	0.91
OTT051685	866	1.00	1.00	1.00	1.00	1.00
OTT061085	891	0.85	0.83	0.86	1.01	0.71
OTT071985	930	0.95	1.32	1.31	1.03	0.66
OTT072685	937	1.18	1.12	0.94	0.94	0.69
OTT080185	943	0.86	0.88	nil.	0.89	0.88
OTT051685[b]	866	7.0%	1.0%	1.0%	2.3%	4.6%

[a]Ratios based on peak height only.

[b]Actual composition determined by QXRD before normalization.

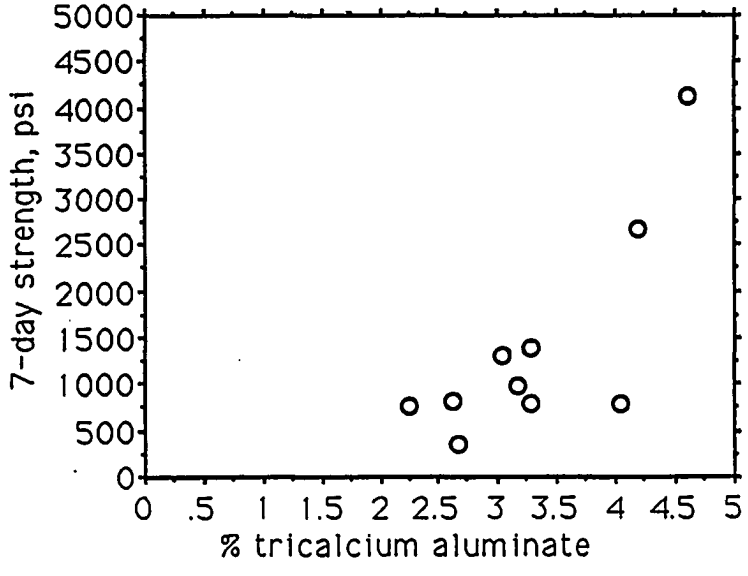


Figure 41. Compressive strength versus tricalcium aluminate content for ten different samples of Ottumwa fly ash

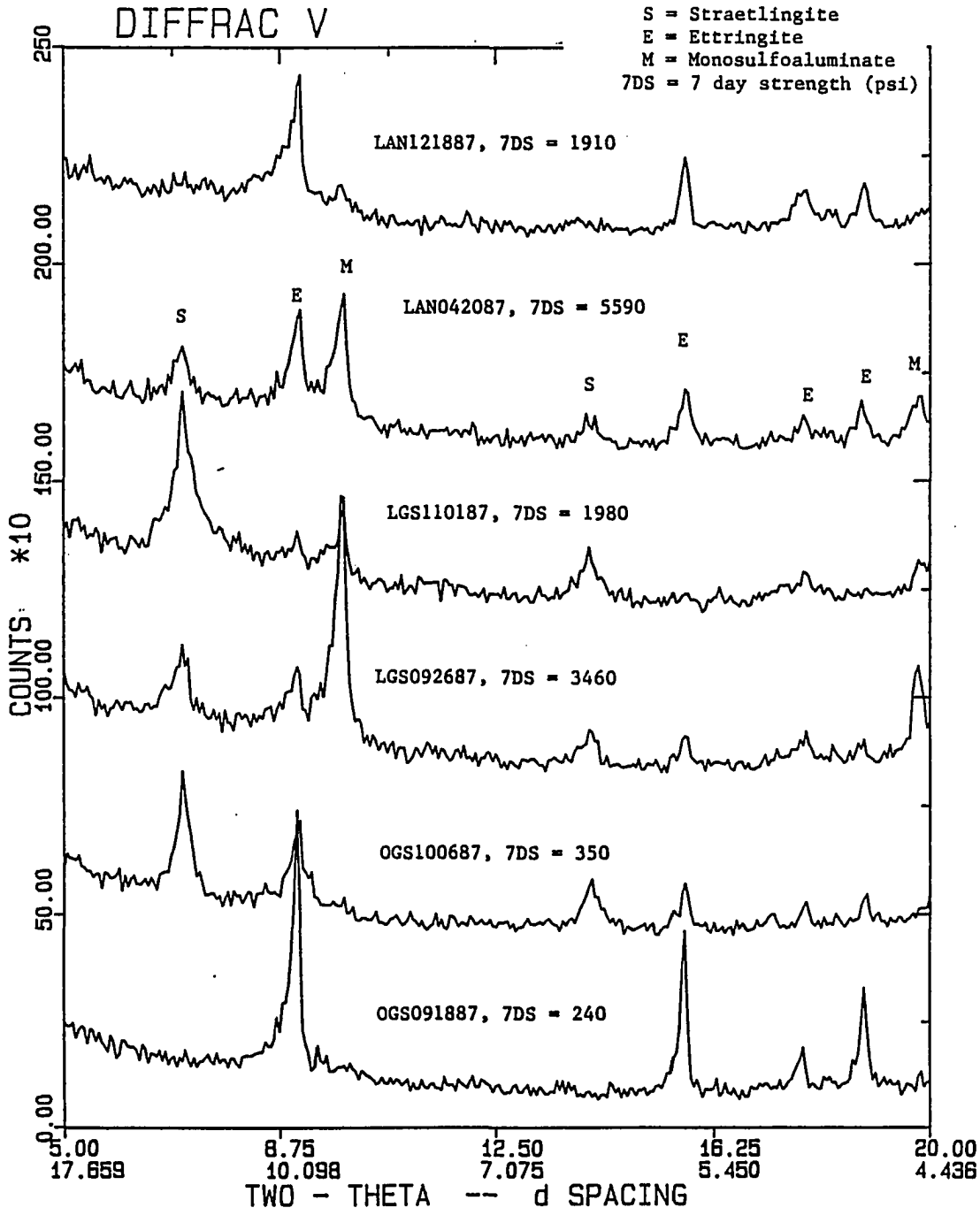


Figure 42. X-ray diffractogram showing the major hydration products commonly observed in Iowa fly ash pastes

relationship between the diffracted intensity of a given compound (i.e., apparent concentration) and compressive strength of the paste specimens. This was not unexpected because other researchers [48, 69] have indicated that quantitative determination of hydration products in cementitious mixtures is a very difficult task. The general trends indicated by this study suggest that the low strength pastes were most commonly associated with ettringite formation, while the high strength pastes were associated with either monosulfoaluminate or straetlingite formation.

The long-term stability of these three hydrates in fly ash pastes appears to be reasonably good. X-ray analysis of paste specimens that had been cured in sealed plastic vials for the past three years indicated that all major phases were still present. Some of the ettringite appeared to have decomposed into monosulfoaluminate during the three-year time period but other changes were minimal.

A Case Study of Ottumwa and Louisa Power Plants

The purpose of this section is to compare fly ash sampled from two very similar power plants before and after a routine maintenance shutdown. Louisa and Ottumwa generating stations (LGS and OGS or OTT, respectively) were chosen for this phase of the study because they burn coal from the same mine, they are similar in size, and they were built and came on line in the early 1980s (see Tables 8 and 9 for additional details). The major difference between the two power plants is that OGS dopes its coal with sodium carbonate while LGS does not (although LGS may have to begin sodium carbonate doping to avoid EPA fines). Hence, we can directly compare fly ash produced from Cordero mine coal with and without sodium carbonate treatment.

The sampling and testing schemes used in this study were described earlier. However, it is pertinent to add that LGS samples were obtained from an autosampler. The LGS autosampler is located between the electrostatic precipitator ash hoppers and the fly ash silo. It samples the ash stream at specific time intervals and produces a one- to two-gallon sample each day. OGS ash samples were taken from ash trucks while loading from a 3000-ton capacity silo. Hence, one must

consider the possibility of silo mixing in the OGS fly ash samples. The OGS fly ash silo was completely emptied during the maintenance outage so fly ash samples taken immediately after start up should reflect transient conditions at the power plant.

Background

The bulk of the MERL fly ash data base consists of information about samples obtained from Ottumwa Generating Station (OGS). Also, OGS personnel have been very receptive to providing power plant operating conditions and maintenance schedules to Iowa State researchers. Hence, the current state of knowledge about the fly ash produced at OGS is well ahead of the other Iowa power plants.

OGS produces about 80,000 tons per year of high-calcium fly ash having a nominal analytical CaO content of about 25%. The power plant burns low sulfur, sub-bituminous coal from the Powder River Basin near Gillette, Wyoming. Sodium carbonate is routinely added to the raw coal feed to enhance the performance of the power plants hot-side electrostatic precipitators.

As mentioned earlier, the compressive strength of Ottumwa fly ash pastes change drastically as a function of sampling date. A plot of the 7-day compressive strength of OGS fly ash pastes versus sampling date is shown in Figure 43. It is evident that the major fluctuations in compressive strength occur during the late spring or late fall months of the calendar year. These fluctuations in compressive strength correspond roughly to the OGS maintenance schedule (note the bars near the x-axis). The lower half of Figure 43 shows the sodium carbonate feed rate, expressed in pounds per ton of coal, that was added to the raw coal to enhance the performance of the electrostatic precipitators. It is apparent that the power plant operating parameters (both sodium carbonate feed rate and routine maintenance periods) influence the strength properties of the OGS fly ash pastes. It must be mentioned that the maintenance cycle is not independent of the sodium carbonate feed rate. In fact, the two are directly related because the sodium carbonate doping is utilized to increase the length of time that the power plant can operate within EPA air

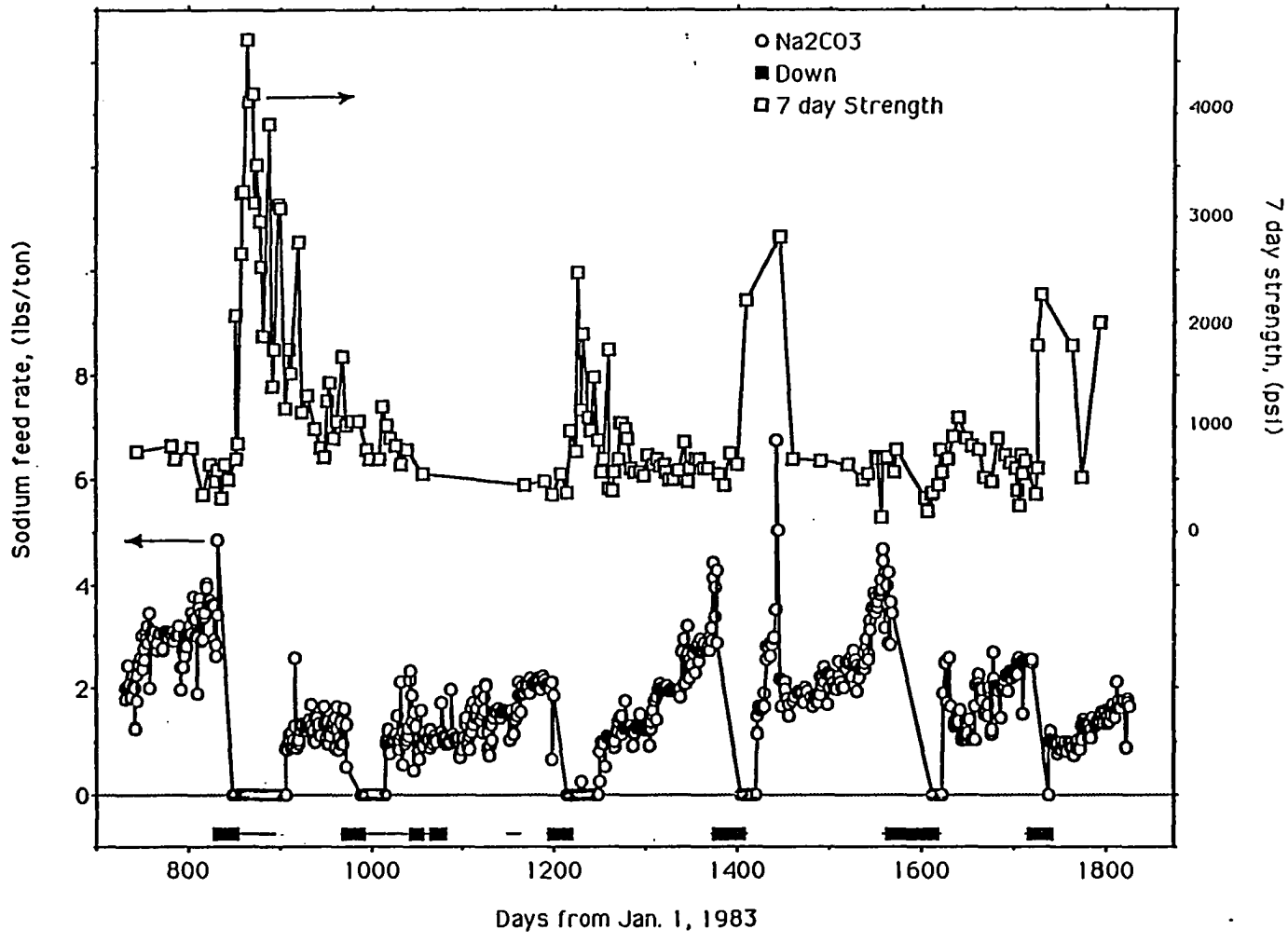


Figure 43. Overlay of the seven-day compressive strength and OGS sodium carbonate feed rate versus sampling date

quality specifications. Hence, the sodium carbonate feed rate is normally cycled during the generating year. After a maintenance shutdown, during which the electrostatic precipitators are washed out, the power plant needs little (or no) sodium carbonate doping to meet EPA specifications. However, when the power plant is approaching a maintenance shutdown, a high sodium carbonate feed rate is normally needed to stay within EPA guidelines. When the sodium carbonate feed rate gets large enough to cause excessive boiler slagging (typically between 3 and 4 pounds of sodium carbonate per ton of raw coal) the power plant will shutdown for cleaning. A plot of the bulk fly ash sodium oxide content versus sampling time is shown in the top portion of Figure 44. The sodium carbonate feed rate is shown in the lower half of Figure 44. As one would expect, the sodium carbonate feed rate used at the power plant directly influences the amount of sodium oxide present in the fly ash. The sulfur trioxide content of the fly ash also exhibited a similar trend; however, it did not correspond to the sodium carbonate feed rate as well as sodium oxide did. The remaining elements monitored in this study (Si, Al, Fe, Mg, Ca, P, and Ti) did not indicate any consistent trends with power plant operating conditions.

OGS versus LGS

Both OGS and LGS fly ashes were sampled about 3 to 4 times per week from early July, 1987 until their scheduled fall maintenance shutdown (actual outages were 9/18/87 through 10/2/87 for OGS, and 9/27/87 through 10/25/87 for LGS). While OGS was off line, samples of fly ash were obtained 3 times per week until the fly ash silo was empty. No ash samples were available while LGS was off line because its autosampler does not function during a shutdown. After start up, ash samples were again obtained from both power plants about 3 or 4 times per week for about 2 weeks. All of the ash samples taken immediately before and after a maintenance shutdown were subjected to chemical analysis, x-ray diffraction analysis and the paste testing scheme. Many of these specimens were also subjected to Blaine fineness testing to monitor the specific surface of the fly ash samples. A sub-group of samples was selected from the remaining ash samples to

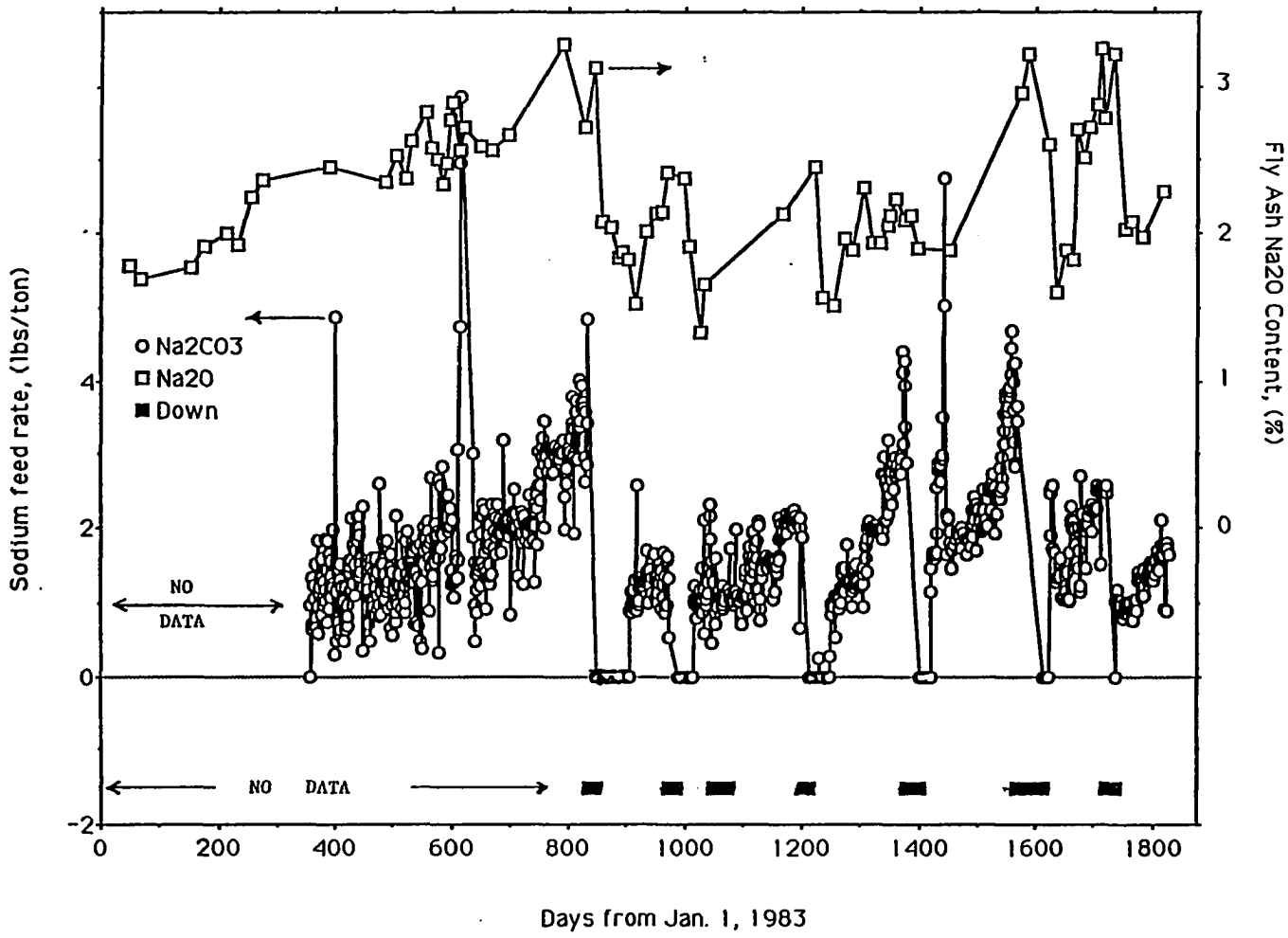


Figure 44. *Overlay of fly ash sodium oxide content and OGS sodium carbonate feed rate versus sampling date*

represent the "background" level of fly ash characteristics that existed before the maintenance shutdown. This sub-group of samples was subjected to the same testing scheme that was described above. Raw data for this phase of the study are summarized in Appendix D.

The results of the paste testing program are summarized in Table 17. In general, the LGS specimens consistently performed better than the OGS specimens in the paste tests. A plot of compressive strength (7-day) versus sampling date is shown in Figure 45. It is interesting to note that the OGS compressive strength tends to increase immediately after start up; this is consistent with the trend reported earlier in this dissertation (see Figure 43). The LGS specimens showed no clear trend, although the compressive strength values were down slightly after start up. Blaine fineness tests indicated only a relatively small change (less than ± 6% from the mean value) in the specific surface of ash samples taken from either power plant. Hence, the fineness of the fly ash does not appear to be playing a major role in determining the bulk physical properties of these fly ash pastes.

Several OGS samples obtained immediately before shutdown and after start up exhibited very anomalous physical properties. None of these samples failed to meet the chemical specifications listed in ASTM C 618. Two of the samples obtained before shutdown (OTT091687 and OTT091887), had severe expansive tendencies when they were removed from the autoclave bar molds. The expansive properties of OGS091687 are illustrated in Figure 46. Please note that the time axis represents the time after the specimen was removed from the mold. The specimen was 1 hour old when it was removed from the mold. The OGS091887 specimen had similar tendencies although they were not as severe (about 0.7% expansion in 4 hours). Both samples had rather high SO_3 contents (4.5% and 3.6% for OGS091687 and OGS091887, respectively) and mineralogical studies indicated that the SO_3 appeared to be present in the fly ash as anhydrite ($CaSO_4$), only small concentrations of tetracalcium trialuminate sulfate were observed. In fact, these two specimens had the highest concentrations of anhydrite that were observed in the OGS

Table 17. Summary of the results of the OGS and LGS paste tests

Test	OGS (n=21) X	S	MAX	MIN	LGS (n=18) X	S	MAX	MIN
Compressive Strength (psi)								
4 hour	307	144	566	33	527	238	936	230
1 day	454	170	793	158	1123	413	1796	368
7 day	799	477	2273	238	2836	743	3893	1267
14 day	940	570	2508	258	3302	949	4499	1593
28 day	1004	584	2277	236	3514	578	4701	2241
56 day	1282	835	3342	229	3754	628	4832	2364
Volume Stability (% Expansion @ 28 Days)[a]								
Air cured	-0.05	0.02	-0.03	-0.08	-0.12	0.05	-0.06	-0.19
Humid cured	0.00	0.01	0.01	-0.02	0.07	0.04	0.15	0.00
Setting Time (minutes)								
Initial set	26	25	97	9	8	2	12	4
Final set	43	50	198	14	11.5	3	19	6
Heat Evolution								
Time to peak (min)	32	13	56	12	29	9	47	18
ΔT (°C)	4	2	7	0.3	4	2	8	2

[a]This statistical summary does not include data from OGS091887 or OGS091687.

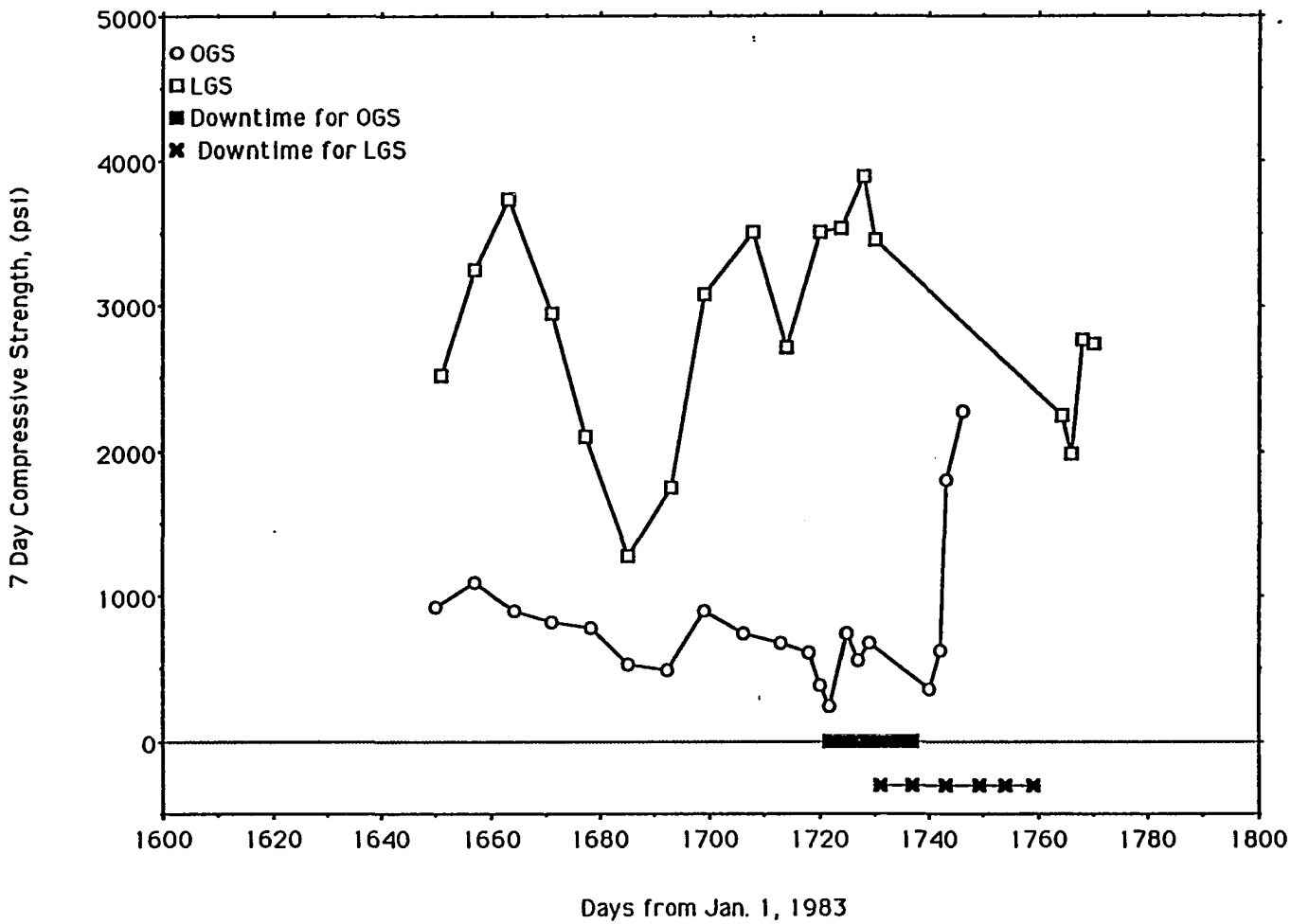


Figure 45. OGS and LGS seven-day paste strengths versus sampling date

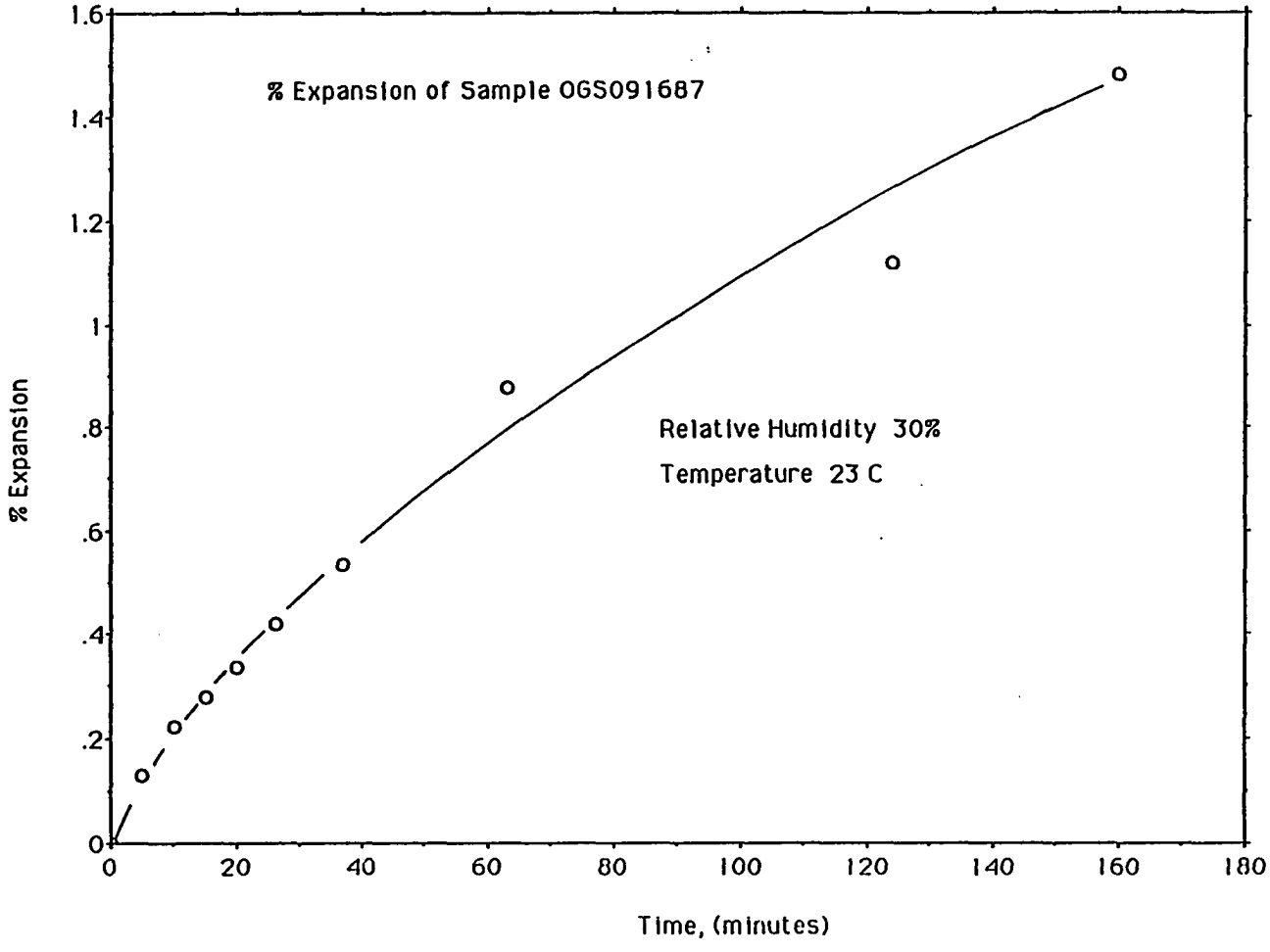


Figure 46. Expansion of OGS091687 versus time

samples during this study. Also, the first two specimens obtained after start up (OGS100687 and OGS100787) had very odd setting and hardening characteristics. Both specimens had final set times of about 3 hours and exhibited poor temperature rise test results. Chemical analysis indicated that the two samples were deficient in analytical CaO and enriched in P_2O_5 (both samples had over 2.2% (by weight) of P_2O_5). Mineralogical studies were in agreement with the chemical studies. XRD indicated that both OGS100687 and OGS100787 were deficient in tricalcium aluminate and free lime. No phosphorous bearing mineral(s) could be identified in the x-ray diffractograms.

Chemical and mineralogical studies of the LGS samples indicated trends similar to those that were observed for the OGS samples. In general, the SO_3 content increased sharply as the power plant neared the maintenance shutdown period (see Figure 47). Mineralogically speaking, this corresponded to an abrupt increase in the amount of anhydrite present in the samples. Again, the concentration of tetracalcium trialuminate sulfate appeared to be nearly constant throughout the study. None of the LGS physical test paste specimens behaved anomalously; however, it is pertinent to add that the two specimens nearest to both shutdown and start up were of such limited quantity that physical tests could not be performed. The sample taken just before shutdown (LGS092787) had a SO_3 content of 6.1%; this sample fails to meet SO_3 specifications in ASTM C 618 specifications. The sample taken immediately after start up (LGS102787) had milo in it. Milo is a grain that is commonly used in place of sand to blast the residue off of the electrostatic precipitator plates during clean out operations. Obviously, the sample containing milo would not meet ASTM C 618 specifications. The ASTM composite sampling procedure would have probably missed rejecting both of these samples because they would have been diluted with four other samples before testing was initiated.

Again, the bulk sodium oxide content of the fly ash appeared to play an important role in the strength development of fly ash pastes. Figure 48 illustrates this fact for the LGS and OGS samples. Eight data points from Lansing fly ash paste tests were included in the figure to help

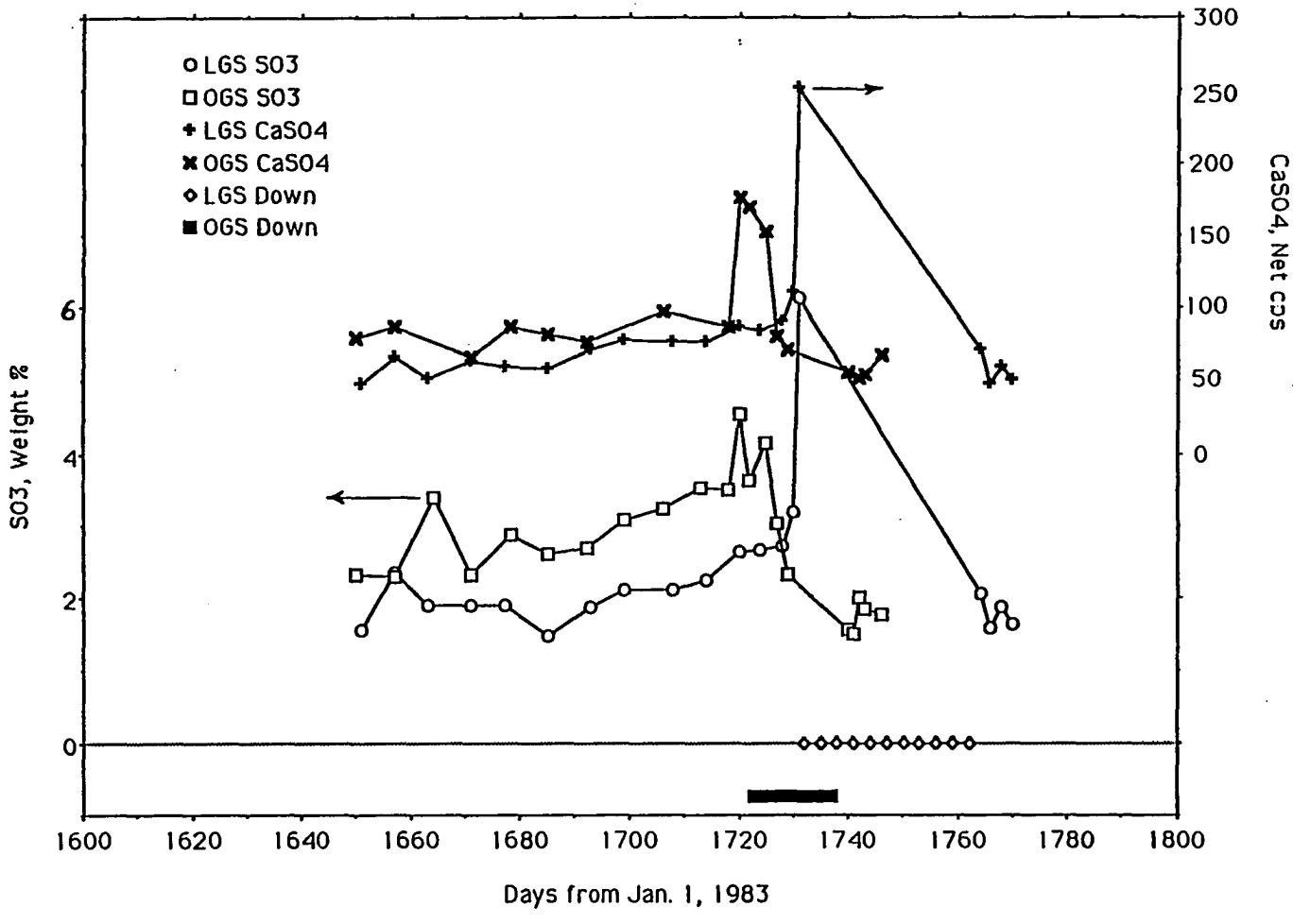


Figure 47. Sulfur trioxide and anhydrite concentrations in LGS and OGS fly ashes as a function of sampling date

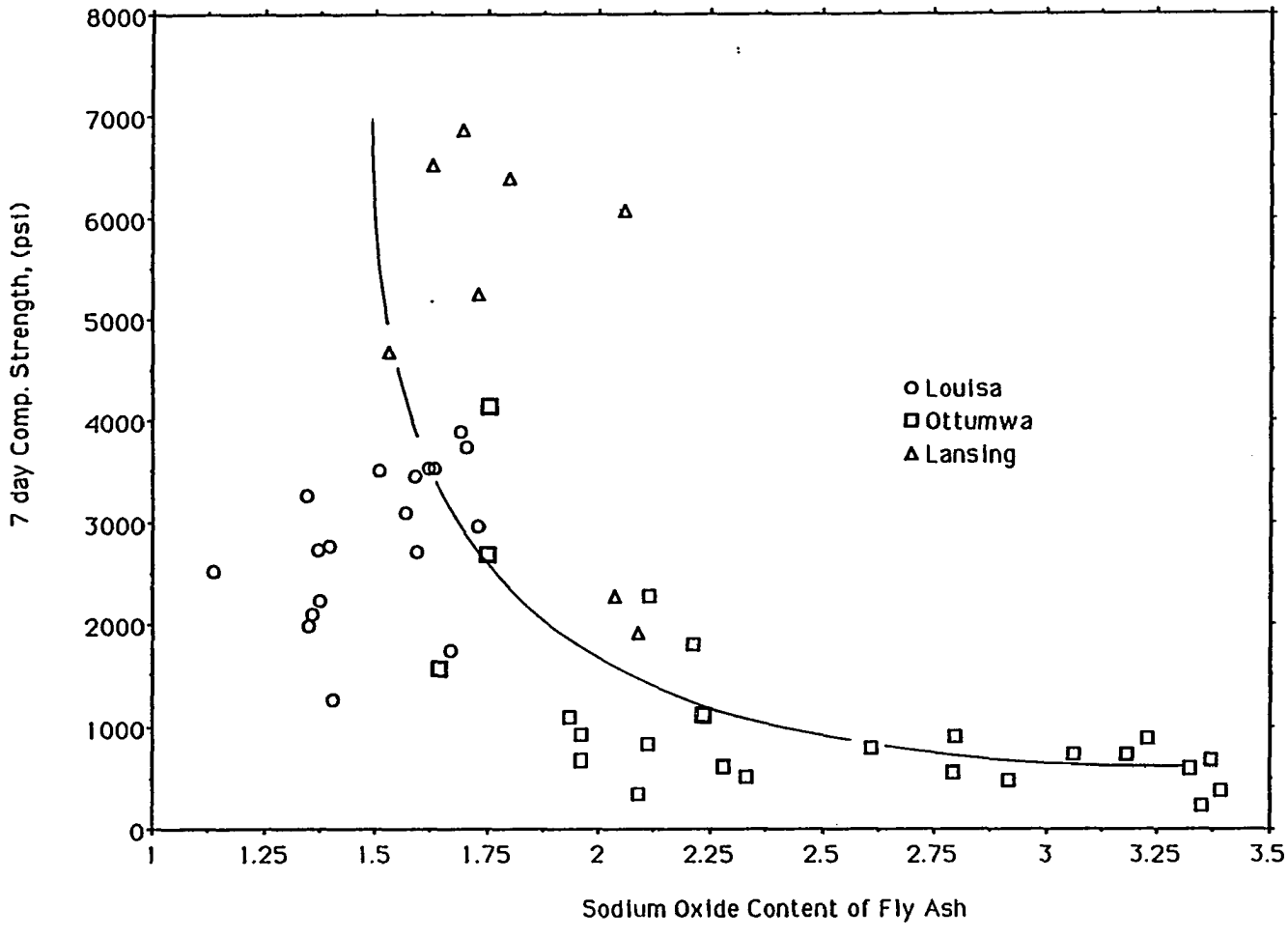


Figure 48. Seven-day compressive strength of fly ash pastes versus sodium oxide content

expand the scale of the vertical axis. The trend line indicated on the figure, was drawn by hand and it does not represent a least-squares fit. In general, when the sodium oxide content of the bulk fly ash exceeds about 2.5% the compressive strength of the paste is reduced. However, one must be very cautious when interpreting Figure 48 because OGS fly ash is the only ash with sodium oxide contents above 2.5%. Also, as was explained earlier, the manner in which the sodium is contained in the fly ash is of extreme importance because different minerals (and/or glasses) contribute different amounts of sodium to the pore solution. A detailed investigation of the pore solution present in the fly ash paste specimens is needed before one can deduce firm conclusions about the influence of alkalis on fly ash pastes.

SUMMARY AND CONCLUSIONS

In summary, a detailed investigation has been made of the physical, chemical and mineralogical characteristics of Iowa high-calcium (Class C) fly ashes. Samples from five Iowa power plants were monitored, as a function of sampling date, to assess the variability of the different ash sources. Fly ash samples obtained during "normal" and "upset" power plant operating conditions were investigated during this study.

ASTM C 311 test methods (with minor modifications) were used to characterize the physical properties (i.e., moisture content, loss on ignition, soundness, fineness, pozzolanic activity and specific gravity) and chemical-physical properties (i.e., bulk chemistry, available alkalis plus the physical tests mentioned earlier) of over 800 fly ash samples; 685 of the ash samples were subjected to physical testing while 189 samples (mostly composite samples) were subjected to chemical-physical testing. About 250 of the physical test samples were also subjected to a paste testing program. The paste tests were used to assess the cementitious characteristics of the various fly ash sources. The paste testing program was also used to identify which of the fly ash samples would be subjected to detailed chemical and mineralogical studies. The results of this research effort, directed toward the development of a rational characterization method for Iowa fly ashes, can be summarized as follows.

1 - The results of ASTM physical and chemical testing, which are commonly used to classify fly ash for use as a mineral admixture in portland cement concrete, show little variation with time, irrespective of ash source. However, part of the reason for the lack of variability in the chemical testing phase of this project can be directly attributed to the ASTM composite sampling scheme. None of the fly ash samples tested during this research project failed to meet ASTM C 618 specifications (this statement ignores two ash samples that were obtained from Louisa Generating Station during shutdown and start up operations).

2 - The available alkali test (described in ASTM C 311) tends to underestimate the amount of alkalis that can be released from Iowa high-calcium fly ashes.

3- The results of the paste testing program indicated that the physical properties of fly ash pastes can change dramatically (by a factor of 5 to 10 in some instances) in short periods of time. The program also linked the power plant maintenance schedule and sodium carbonate coal pre-treatment at Ottumwa generating station, to cyclical trends in fly ash paste strength properties. Fly ash properties (both chemical and physical) generally change drastically immediately before or after a maintenance outage.

4- Strong correlations were observed between several of the variables studied in the fly ash paste testing program. The most obvious correlations were between 7-day and 28-day compressive strengths, between compressive strength and temperature rise, and between initial and final set.

5- Fly ash paste mixes exhibited significant differences in volume stability characteristics depending on the mode of curing (i.e., air curing or humid curing). However, in most instances the shrinkage/expansive tendencies of the fly ash pastes were not severe.

6- X-ray diffraction analysis indicated that all of the fly ashes contained the same major crystalline compounds plus a significant portion of glassy material. The crystalline compounds identified in the fly ashes were: lime, periclase, alpha-quartz, anhydrite and a mineral very similar to tricalcium aluminate. Many of the fly ashes also contained tetracalcium trialuminate sulfate and a ferrite spinel. The concentrations of these crystalline compounds changed significantly in ashes sampled from the various power plants, they also changed in samples taken from a single power plant at different sampling times. Hence, mineralogy appears to play a very important role in determining the physical properties of fly ash pastes. Two different types of glass were found in the various fly ashes. The major glass type appears to consist mostly of calcium, aluminum and silicon; this glass was soluble in hydrochloric acid. The minor glass type found in the fly ashes was nearly insoluble in hydrochloric acid; this glass was very similar to those that are commonly found in Class F fly ashes (i.e., more siliceous in character).

7- Both bulk mineralogy and bulk chemistry were found to depend heavily on the particle size fraction of a given fly ash that was being investigated. Typically, the

alkaline earth elements (Ca, Mg, Sr and Ba) tended to accumulate in the smaller particle size fractions at the expense of Si. Mineralogically, anhydrite and the calcium aluminate silicate glass phase were enriched in the smaller particle size fractions at the expense of alpha-quartz.

8- Chemical, mineralogical and physical testing indicated that sodium, the sulfate bearing minerals (or bulk SO_3), lime and tricalcium aluminate contents of the fly ashes all appeared to play important roles in the development of hydration products in the paste specimens. All of the fly ash paste specimens studied in this research project contained similar reaction (hydration) products. The fly ash pastes that exhibited high compressive strengths normally contained monosulfoaluminate and straetlingite as the major hydration products, along with minor amounts of ettringite. The weak fly ash pastes always contained ettringite as a major (often only) constituent. The weak pastes occasionally contained lesser amounts of monosulfoaluminate and straetlingite. The exact link between chemistry, mineralogy and the physical properties of fly ash pastes have not been exactly defined by this research.

On the basis of the findings described above, one must conclude that the ASTM test methods and specifications are not sufficient for a complete and diagnostic classification of Iowa high-calcium fly ashes. Based on the correlations established in this research, relatively simple and short term diagnostic test methods (such as paste strength, temperature rise or time of set) can be used to help the current classification scheme to more effectively differentiate between high-calcium Iowa fly ashes. More effective categorization of Iowa fly ashes should ultimately lead to more effective utilization of our fly ash resources in the years to come.

CLOSING COMMENTS

Throughout this dissertation very few attempts have been made to designate whether a given fly ash is "good" or "bad". Instead, this author has stressed the characterization of the chemical and physical properties of fly ash. This was done so that innovative people could ascertain whether they could attempt to use high-calcium fly ash in place of other cementitious materials. Too often people turn to existing ASTM specifications to decide if a fly ash can be used, and unfortunately, as was discussed earlier in this dissertation, the ASTM criteria are not appropriate for many situations. Hence, many designers still shun the use of fly ash. This must change. Obviously, there are "good" and "bad" fly ashes; however, one must remember that what is deemed as "bad" for one use may be "good" for another, or vice-versa. Hence, to increase the use of fly ash many processes (both existing and new) will have to rely on performance specifications rather than design specifications (also referred to as "prescriptive" specifications earlier in this dissertation).

Recommendations

Fly ash as an admixture

First, the current composite sampling scheme (described in ASTM C 618) needs to be re-evaluated. The composite sampling scheme appeared to reduce the variability of the physical properties of the fly ashes studied in this research. This effect becomes magnified as a given power plant approaches (or exits) a routine maintenance outage. In extreme situations one may expect that one or more of the individual samples that are combined to form a composite sample, may not meet the ASTM C 618 specifications. Hence, it is recommended to increase the sampling frequency as a power plant approaches an outage, and the samples should be analyzed individually, not as composite samples.

Second, the moisture content test should be removed from the physical and chemical-physical test specifications for high-calcium fly ashes. These fly ashes tend to react quickly with water to form hydration products. Hence, they do not contain free water. A bulk loss on ignition test similar to the one currently used for portland cement would be a logical replacement for the existing test.

Finally, the available alkali test should be removed from the ASTM chemical testing specifications for fly ash. This test does not accurately indicate the amount of alkalis that can be released from fly ashes. The available alkali test could be replaced with an alternative test that measures total alkalis and/or water soluble alkalis.

Fly ash as a cement

The fly ashes investigated in this dissertation behaved similarly to hydraulic cements. Hence, there is great potential for the use of these fly ashes as stabilization agents (or drying agents) in the construction industry. Also, such fly ashes exhibit great potential for encapsulation of hazardous wastes or as a raw material for manufacturing artificial aggregates. The compressive strength, volume stability and setting time of fly ash-water mixtures are the pertinent physical properties that govern the use of fly ash in these situations. These properties can be measured fairly easily by using the test methods (or similar methods) described in this dissertation. Performance specifications for a given fly ash can be devised for many applications by extrapolating the laboratory test results to field situations. It will be imperative to monitor the field applications, both during construction and over a significant period of time, to provide a firm basis for updating the performance specifications.

The chemistry and mineralogy of a given fly ash definitely influence its physical properties. However, this dissertation has done very little to provide a concise link between the chemical and physical properties of fly ash. This is an area of fly ash research that holds great potential for future

investigators. The literature abounds with information concerning the physical and chemical properties of an enormous variety of fly ashes. This author believes that the time is ripe for investigating the relationships between the physical and chemical properties of fly ash. Such a relationship could be generalized mathematically as listed below.

$$P = A\Sigma w \cdot C + B\Sigma w \cdot G + D(S)$$

where: **P** is any property of fly ash (e.g., specific gravity, compressive strength, etc.)

A,B,D are arbitrary coefficients

w represents the weight fraction of a given constituent

C represents the concentration of a given crystalline component

G represents the concentration of a given type of glass

S represents the specific surface (or fineness) of the fly ash

The assertion that the property term (P) is linearly related to the remaining terms is purely speculative at this time. However, it does provide a simple model that can be subjected to rigorous testing. Similar models have been of great importance to the glass industry where they they have simplified the estimation of the pertinent properties of glasses with different compositions. In the case of fly ash, the addition of the specific surface and crystalline component terms should help to extend the model so that it can be applied to a large number of different fly ashes. The model also stresses the fact that fly ash chemistry (i.e., the combination of bulk chemical analysis with mineralogical information) can no longer be ignored.

REFERENCES

1. Abdun-Nur, E. A. "Fly Ash in Concrete—An Evaluation." *Highway Research Board* Bulletin 284, 1961.
2. Adriano, D. C. *Trace Elements in the Terrestrial Environment.* New York: Springer-Verlag, 1986.
3. Aitcin, P. C., F. Autefage, A. Carles-Gibergues, and A. Vaguier. "Comparative Study of the Cementitious Properties of Different Fly Ashes." *ACI Special Publication* No. 91, 1986.
4. American Society for Testing and Materials. *1988 Annual Book of ASTM Standards.* Vol. 4.01. Philadelphia, PA: ASTM, 1988.
5. American Society for Testing and Materials. *1988 Annual Book of ASTM Standards.* Vol. 4.02. Philadelphia, PA: ASTM, 1988.
6. American Society for Testing and Materials. *1988 Annual Book of ASTM Standards.* Vol. 5.05. Philadelphia, PA: ASTM, 1988.
7. *Annual Energy Review 1988.* DOE/EIA-0384(89). Washington, D.C.: U.S.D.O.E., May 1989.
8. Bergeson, Kenneth L. "The Glassy Phase and Reaction Products of Iowa Fly Ashes." Ph. D. Dissertation, Iowa State University, 1986.
9. Berry, E. E., R. T. Hemmings, and D. M. Golden. "Enhanced Resource Recovery by Benefication and Direct Acid Leaching of Fly Ash." In *Fly Ash and Coal Conversion By-Products: Characterization, Utilization, and Disposal III,* Vol. 86. Eds. G. J. McCarthy, F. P. Glasser, D. M. Roy, and S. Diamond. Pittsburgh, PA: Materials Research Society, 1987. 365-380.

10. Berry, E. E., R. T. Hemmings, and B. J. Cornelius. "Speciation in Size and Density Fractionated Fly Ash III. The Influence of HCl Leaching on the Glassy Constituents of a High-Ca Fly Ash." In *Fly Ash and Coal Conversion By-Products: Characterization, Utilization, and Disposal IV,* Vol. 113. Eds. G. J. McCarthy, F. P. Glasser, D. M. Roy, and R.T. Hemmings. Pittsburgh, PA: Materials Research Society, 1988. 55-63.

11. Biggs, D. L. and J. J. Bruns. "Transmitted and Reflected Visible Light Microscopy of Two Bituminous Fly Ashes." In *Fly Ash and Coal Conversion By-Products: Characterization, Utilization, and Disposal I,* Vol. 43. Eds. G. J. McCarthy, and R.J. Lauf. Pittsburgh, PA: Materials Research Society, 1985. 21-29.

12. Brink, R. H. and W. J. Halstead. "Studies Related to the Testing of Fly Ash for Use in Concrete." *Proceedings of the American Society for Testing and Materials* **56** (1956): 1161-1206.

13. Burnet, G., M. J. Murtha, and J. W. Dunker. *Recovery of Metals from Coal Ash—An Annotated Bibliography.* U.S.D.O.E.—IS4833 (rev): UC-90. Ames Laboratory, Ames, IA, 1984.

14. Butler, W. B. "A Critical Look at ASTM C618 and C311." *Cement, Concrete, and Aggregates* **4** (1982): 68-72.

15. Butler, W. B. and M. A. Mearing. "Fly Ash Benefication and Utilization in Theory and Practice." In *Fly Ash and Coal Conversion By-Products: Characterization, Utilization, and Disposal II,* Vol. 65. Eds. G. J. McCarthy, F. P. Glasser and D. M. Roy. Pittsburgh, PA: Materials Research Society, 1986. 11-17.

16. Chopra, S. K. and S. N. Narain. "Measurement of the Specific Surface of Fly Ash." *Materials Research and Standards* **4** (1964): 487-490.

17. *Classification of Fly Ash for Use in Cement and Concrete.* Electric Power Research Institute CS-5516. Final Report for Project 2422-10. E.P.R.I., Palo Alto, California, April 1987.

18. *Combustion—Fossil Power Systems.* 3rd edition. Ed. J. E. Singer. Windsor, CT: Combustion Engineering, Inc., 1981.

19. Davis, Raymond E., R. W. Carlson, J. W. Kelley, and H. E. Davis. "Properties of Cements and Concretes Containing Fly Ash." *Proceedings of the American Concrete Institute* **33** (May-June 1937): 577-612.

20. Davis, Raymond E., H. E. Davis, and J. W. Kelley. "Weathering Resistance of Concretes Containing Fly Ash Cements." *Proceedings of the American Concrete, Institute* **37** (January 1941): 281-293.

21. Diamond, S. "On the Glass Present in Low-Calcium and High-Calcium Fly Ashes." *Cement and Concrete Research* **13** (1983): 459-464.

22. Diamond, S. "The Characterization of Fly Ashes." In *Proceedings, Symp. N, Effects of Fly Ash Incorporation in Cement and Concrete.* Editor S. Diamond. University Park, PA: Materials Research Society, 1982. 12-23.

23. Eberendu, A. N. R. "The Quantitative Determination of Glass in Slag and Fly Ash by Infrared Spectroscopy." Ph. D. Dissertation, North Texas State University, Denton, TX, 1983.

24. Fisher, G. L., B. A. Prertice, D. Silberman, J. M. Ondov, A. H. Bierman, R. C. Ragaini, and A. R. McFarland. "Physical and Morphological Studies of Size-Classified Coal Fly Ash." *Environmental Science and Technology* **12** (1978): 447-451.

25. Furuya, K., Y. Miyajima, T. Chiba, and T. Kikuchi. "Elemental Characterization of Particle Size-Density Separated Coal Fly Ash by Spectrophotometry, ICPES, and Scanning Electron Microscopy—Energy Dispersive X-ray Analysis." *Environmental Science and Technology* **21** (1987): 898-903.

26. Gilpin, A. *Dictionary of Fuel Technology.* New York, New York: Philosophical Library Inc., 1969.

27. Glasser, F. P., S. Diamond, and D. M. Roy. "Hydration Reactions in Cement Pastes Incorporating Fly Ash and Other Puzzolanic Materials." In *Fly Ash and Coal Conversion By-Products: Characterization, Utilization, and Disposal III*, Vol. 86. Eds. G. J. McCarthy, F. P. Glasser, D. M. Roy, and S. Diamond. Pittsburgh, PA: Materials Research Society, 1987. 139-158.

28. Gooch, J. P., E. B. Dismukes, R. E. Bickelhaupt, and R. F. Altman. "Flue Gas Conditioning Studies." *Proceedings of the 2nd International Conference on Electrostatic Precipitators*, Nov. 1984, Koyoto, Japan. Seinichi Masuda, Editor. Pittsburgh, PA: Air Pollution Control Association, 1985. 200-209.

29. Harris, Harry A., J. L. Thompson, and T. E. Murphy. "Factors Affecting the Reactivity of Fly Ash from Western Coals. *Cement, Concrete, and Aggregates* 9 (1987): 34-37.

30. Helmuth, R. *Fly Ash in Cement and Concrete.* Skokie, IL: Portland Cement Association, 1987.

31. Hemmings, R. T. and E. E. Berry. "Speciation in Size and Density Fractionated Fly Ash." In *Fly Ash and Coal Conversion By-Products: Characterization, Utilization, and Disposal II*, Vol. 65. Eds. G. J. McCarthy, F. P. Glasser and D. M. Roy. Pittsburgh, PA: Materials Research Society, 1986. 91-104.

32. Hemmings, R. T. and E. E. Berry. "On the Glass in Coal Fly Ashes: Recent Advances." In *Fly Ash and Coal Conversion By-Products: Characterization, Utilization, and Disposal IV*, Vol. 113. Eds. G. J. McCarthy, F. P. Glasser, D. M. Roy, and R. T. Hemmings. Pittsburgh, PA: Materials Research Society, 1988. 3-38.

33. Hemmings, R. T., E. E. Berry, B. J. Cornelius, and B. E. Scheetz. "Speciation in Size and Density Fractionated Fly Ash II. Characterization of a Low-Calcium, High-Iron Fly Ash." In *Fly Ash and Coal Conversion By-Products: Characterization, Utilization, and Disposal III*, Vol. 86. Eds. G. J. McCarthy, F. P. Glasser, D. M. Roy, and S. Diamond. Pittsburgh, PA: Materials Research Society, 1987. 81-98.

34. Hime, W. G. "ASTM Method for the Surface Area Analysis of Portland Cement." In *Fineness of Cement*, ASTMSTP473. Philapelphia, PA: American Society for Testing and Materials, 1970. 3-19.

35. Holcombe, L. J., B. P. Eynon, and P. Switzer. "Variability of Elemental Concentrations in Power Plant Ash." *Environmental Science and Technology* 19 (1985): 615-620.

36. Isenberger, K. "Report on Fly Ash Variability." Highway Division, Office of Materials, Iowa Dept. of Transportation, Ames, Iowa, March, 1981.

37. Joshi, R. C. and B. K. Marsh. "Some Physical, Chemical and Mineralogical Properties of Some Canadian Fly Ashes." In *Fly Ash and Coal Conversion By-Products: Characterization, Utilization, and Disposal III*, Vol. 86. Eds. G. J. McCarthy, F. P. Glasser, D. M. Roy, and S. Diamond. Pittsburgh, PA: Materials Research Society, 1987. 113-125.

38. Katz, J. *The Art of Electrostatic Precipitation.* Pittsburgh, PA: S&S Printing Co., Inc., 1979.

39. Kaufhen, N., M. Sherasa, and D. Lichtman. "X-ray Photoelectron Spectroscopy Studies of Coal Fly Ashes with Emphasis on Depth Profiling of Submicrometer Particle Size Fractions." *Environmental Science Technology* 19 (1985): 609-614.

40. Kaushal, S., D. M. Roy, and P. H. Licastro. "Monitoring of Temperatures in Cementitious Monoliths." In *Fly Ash and Coal Conversion By-Products: Characterization, Utilization, and Disposal III*, Vol. 86. Eds. G. J. McCarthy, F. P. Glasser, D. M. Roy, and S. Diamond. Pittsburgh, PA: Materials Research Society, 1987. 265-275.

41. *Keystone Coal Industry Manual.* Editor C. C. Calhoun. New York, NY: McGraw-Hill (1987).

42. Kilgour, C. L. and S. Diamond. "The Internal Structure of a Low-Calcium Fly Ash." In *Fly Ash and Coal Conversion By-Products: Characterization, Utilization, and Disposal IV*, Vol. 113. Eds. G. J. McCarthy, F. P. Glasser, D. M. Roy, and S. Diamond. Pittsburgh, PA: Materials Research Society, 1987. 65-74.

43. Kirshenbaum, H. "The Classical Chemical Analysis of Silicate Rocks—The Old and the New." *Geological Survey Bulletin 1547.* Washington, D.C.: U. S. Government Printing Office, 1983.

44. Kolthoff, I. M. and E. B. Sandell. *Textbook of Quantitative Inorganic Analysis.* 3rd edition. New York, NY: Macmillan Company, 1952.

45. Kuzyk, P. "Use of Conductance to Monitor Fly Ash Hydration." Master's Thesis, Iowa State University, 1987.

46. Lea, F. M. and C. H. Deusch. *The Chemistry of Cement and Concrete.* New York, NY: St. Martin's Press, Inc., 1956.

47. Lee, Chau, S. Schlorholtz, and T. Demirel. "Available Alkalis in Fly Ash." In *Fly Ash and Coal Conversion By-Products: Characterization, Utilization, and Disposal II*, Vol. 65. Eds. G. J. McCarthy, F. P. Glasser and D. M. Roy. Pittsburgh, PA: Materials Research Society, 1986. 125-130.

48. Locher, F. W. "Hydraulic Properties and Hydration of Glasses of the System CaO-Al_2O_3-SiO_2." *Fourth International Symposium on the Chemistry of Cement*, Monograph No. 43. Washington, D.C.: U. S. National Bureau of Standards, 1960. 267-276.

49. Majling, J., V. Jesenak, D. M. Roy, and R. Roy. "Generalized Bogue Calculations for Determining Phase Compatibility/Equilibria: Assessing Potential Use of Inorganic Industrial By-Products," *Journal of Materials Research* 4 (1989): 447-451.

50. Manz, Oscar E. "Review of International Specifications for Use of Fly Ash in Portland Cement Concrete." *ACI Special Publication* 79 (1983): 189-200.

51. Manz, Oscar E. "Proposed Revisions to Specifications and Test Methods for Use of Fly Ash in Portland Cement Concrete." *ACI Special Publication* **91** (1986): 659-680.

52. McCarthy, G. J. "X-ray Powder Diffraction for Studying the Mineralogy of Fly Ash." In *Fly Ash and Coal Conversion By-Products: Characterization, Utilization, and Disposal IV*, Vol. 113. Eds. G. J. McCarthy, F. P. Glasser, D. M. Roy, and R.T. Hemmings. Pittsburgh, PA: Materials Research Society, 1988. 75-86.

53. McCarthy, G. J. and A. Thedchanamoorthy. "Semiquantitative X-ray Diffraction Analysis of Fly Ash by the Reference Intensity Ratio Method." In *Fly Ash and Coal Conversion By-Products: Characterization, Utilization, and Disposal V*, Vol. 136. Eds. R.T. Hemmings, E.E. Berry, G. J. McCarthy and F. P. Glasser. Pittsburgh, PA: Materials Research Society, 1989. 67-76.

54. McCarthy, G. J., O. E. Manz, D. M. Johansen, S. J. Steinward, and R. J. Stevenson. "Correlations of Chemistry and Minerology of Western U.S. Fly Ash." In *Fly Ash and Coal Conversion By-Products: Characterization, Utilization, and Disposal III*, Vol. 86. Eds. G. J. McCarthy, F. P. Glasser, D. M. Roy, and S. Diamond. Pittsburgh, PA: Materials Research Society, 1987. 109-112.

55. McCarthy, G. J., K. D. Swanson, L. P. Keller, and W. C. Blatter. "Minerology of Western Fly Ash." *Cement and Concrete Research* **14** (1984): 471-478.

56. McKevall, W. C. and W. B. Ledbetter. "Variability and Control of Class C Fly Ash." *Cement, Concrete, and Aggregates* **4** (1982): 87-93.

57. Mehta, P. K. "Pozzolanic and Cementitiuos By-Products as Mineral Admixtures for Concrete—A Critical Review. *ACI Special Publication* **79** (1983): 637-660.

58. Mehta, P. K. "Influence of Fly Ash Characteristics on the Strength of Portland-Fly Ash Mixtures." *Cement and Concrete Research* **15** (1985): 669-674.

59. Mehta, P. K. "Standard Specifications for Mineral Admixtures—An Overview." *ACI Special Publication* **91** (1986): 637-658.

60. Mielenz, R. C. "Specifications on Fly Ash for Use in Concrete: Where We Are—Where Should We Go?" In *Proceedings of the Fifth International Ash Symposium.* Eds. J. S. Spencer and C. E. Whieldon. Morgantown, WV: United States Department of Energy 1979. 482-495.

61. Mills, R. H. "Characterization of Fly Ash Hydration Products From Microstructure." *Fly Ash and Coal Conversion By-Products: Characterization, Utilization, and Disposal II*, Vol. 65. Editors G. J. McCarthy, F. P. Glasser, and D. M. Roy. Pittsburgh, PA: Materials Research Society, 1986. 207-217.

62. Mindess, S. and J. F. Young. *Concrete.* Englewood Cliffs, NJ: Prentice-Hall Inc., 1981.

63. Mings, M. L. "Effect of Fly Ash Reactivity on Fly Ash Paste." Master's Thesis, Iowa State University, 1982.

64. Mings, M. L., S. Schlorholtz, J. M. Pitt, and T. Demirel. "Characterization of Fly Ash by X-ray Analysis Methods." *Transportation Research Record* 941 (1983): 5-11.

65. Minnick, L. J. "Investigations Relating to the Use of Fly Ash as a Pozzolanic Material and as an Admixture in Portland Cement Concrete." *Proceedings of the American Society for Testing and Materials* 54 (1954): 1129-1158.

66. Minnick, L. J. , W. C. Webster and E. J. Purdy. "Prediction of the effect of fly ash in portland cement mortar and concrete." Journal of Materials, 6 (1971): 63-187.

67. Morse, J. G. "Use of Fly Ash in Cement: An Analysis of Potential Environmental Problems." *Colorado School of Mines Mineral Industries Bulletin* 22 (May 1979): 1-12.

68. Neville, M. and A. F. Sarofim. "The Fate of Sodium During Pulverized Coal Combustion." *Fuel* 64 (1985): 387-390.

69. Odler, I. and S. Abdul-Maula. "Possibilities of Quantitative Determination of the AFt-(Ettringite) and AFm-(Monosulfate) Phases in Hydrated Cement." *Cement and Concrete Research* 14 (1984): 133-141.

70. Odler, I. and K. H. Zysk. "Investigations on the Composition of Individual Fly Ash and CFB-Ash Particles." In *Fly Ash and Coal Conversion By-Products: Characterization, Utilization, and Disposal V,* Vol. 136. Eds. R.T. Hemmings, E.E. Berry, G. J. McCarthy and F. P. Glasser. Pittsburgh, PA: Materials Research Society, 1989. 131-137.

71. Pitt, J. M., M. L. Mings, and S. Schlorholtz. "Characterization and Techniques for Rapid Evaluation of Iowa Fly Ashes." *Transportation Research Record* 941 (1983): 12-17.

72. Pitt, J. M., T. Demirel, S. Schlorholtz, R. Hammerberg, and R. Allenstein. "Final Report: Characterization of Fly Ash for Use in Concrete." Iowa Highway Research Board (Ames, IA) Project HR-225, September 1983.

73. Price, Walter H. "Pozzolans—A Review." *ACI Journal* 72 (1975): 225-232.

74. Qian, J. C. and F. P. Glasser. "Bulk Composition of the Glassy Phase in Some Commercial PFA's." In *Fly Ash and Coal Conversion By-Products: Characterization, Utilization, and Disposal IV,* Vol. 113. Eds. G. J. McCarthy, F. P. Glasser, D. M. Roy, and R.T. Hemmings. Pittsburgh, PA: Materials Research Society, 1988. 339-44.

75. Qian, J. C., E. E. Lachowski, and F. P. Glasser. "Microstructure and Chemical Variation in Class F Fly Ash Glass." In *Fly Ash and Coal Conversion By-Products: Characterization, Utilization, and Disposal IV,* Vol. 113. Eds. G. J. McCarthy, F. P. Glasser, D. M. Roy, and R.T. Hemmings. Pittsburgh, PA: Materials Research Society, 1988. 45-53.

76. Qian, J. C., E. E. Lachowski, and F. P. Glasser. "The Microstructure of National Bureau of Standards Reference Fly Ashes." *Fly Ash and Coal Conversion By-Products: Characterization, Utilization, and Disposal V,* Vol. 136. Editors R. T. Hemmings, E. E. Berry, G. J. McCarthy, and F. P. Glasser. Pittsburgh, PA: Materials Research Society, 1989. 77-85.

77. Raask, E. *Mineral Impurities in Coal Combustion.* New York, NY: Hemisphere Publishing Corporation, 1985.

78. Ravina, D. "Production and Collection of Fly Ash for Use in Concrete." In *Proceedings, Symp. N, Effects of Fly Ash Incorporation in Cement and Concrete.* Ed. S. Diamond. University Park, PA: Materials Research Society, 1982. 2-11.

79. Roode, Mark Van, E. Douglas, and R. T. Hemmings. "X-ray Diffraction Measurement of Glass Content in Fly Ash and Slags." *Cement and Concrete Research* **17** (1987): 189-197.

80. Rossouw, Eddie and J. Kruger. "Review of Specifications for Additions for Use in Concrete." *ACI Special Publication* **79** (1983).

81. Scheetz, B. E., D. W. Strickler, M. W. Grutzeck, and D. M. Roy. "Physical and Chemical Behavior of Selectively Etched Fly Ashes." In *Proceedings, Symposium N, Effects of Fly Ash Incorporation in Cement and Concrete.* Editor S. Diamond. University Park, PA: Materials Research Society, 1981. 24-33.

82. Scheetz, Barry E. and W. B. White. "Characterization of Crystalline Phases in Fly Ash by Microfocus Raman Spectroscopy." In *Fly Ash and Coal Conversion By-Products: Characterization, Utilization, and Disposal I,* Vol. 43. Eds. G. J. McCarthy and R. L. Lauf. Pittsburgh, PA : Materials Research Society, 1985. 53-60.

83. Schlorholtz, S. and M. Boybay. "Quantitative X-ray Fluorescence Analysis for Fly Ash Samples." *Advances in X-ray Analysis* **27** (1984): 497-504.

84. Schlorholtz, S. and T. Demirel. "A Comparison of Several Sample Preparation Techniques for the Analysis of Fly Ash." *Advances in X-ray Analysis* **30** (1987): 251-256.

85. Schlorholtz, S., K. Bergeson, and T. Demirel. "Variability and Trends in Iowa Fly Ashes." In *Fly Ash and Coal Conversion By-Products: Characterization, Utilization, and Disposal III,* Vol. 86. Eds. G. J. McCarthy, F. P. Glasser, D. M. Roy, and S. Diamond. Pittsburgh, PA: Materials Research Society, 1987. 325-336.

86. Schlorholtz, S., K. Bergeson, and T. Demirel. "Monitoring of Fluctuations in the Physical Properties of a Class C Fly Ash." In *Fly Ash and Coal Conversion By-Products: Characterization, Utilization, and Disposal III*, Vol. 86. Eds. G. J. McCarthy, F. P. Glasser, D. M. Roy, and S. Diamond. Pittsburgh, PA: Materials Research Society, 1987. 337-342.

87. Schlorholtz, S., K. Bergeson, and T. Demirel. "Monitoring of Fluctuations in the Physical and Chemical Properties of a High-Calcium Fly Ash." In *Fly Ash and Coal Conversion By-Products: Characterization, Utilization, and Disposal IV*, Vol. 113. Eds. G. J. McCarthy, F. P. Glasser, D. M. Roy, and R.T. Hemmings. Pittsburgh, PA: Materials Research Society, 1988. 107-116.

88. Schlorholtz, S., T. Demirel, and J. M. Pitt. "An Examination of the ASTM Lime Pozzolanic Activity Test for Class C Fly Ashes." *Cement and Concrete Research* **14** (1984): 499-504.

89. Shannon, R. H. *Handbook of Coal-Based Electric Power Generation.* Park Ridge, NJ: Noyes Publications, 1982.

90. Simmons, H. S. and J. W. Jeffery. "An X-ray Study of Pulverized Fuel Ash." *Journal of Applied Chemistry* **10** (1960): 328-336.

91. Smock, R. "Hot-side Precipitator Problems Plague Western Coal Burners." *Electric Light and Power*, Oct., 1980, pp. 37-38.

92. Stevenson, R. J. and T. P. Huber. "SEM Study of Chemical Variations in Western U.S. Fly Ash." In *Fly Ash and Coal Conversion By-Products: Characterization, Utilization, and Disposal III*, Vol. 86. Eds. G. J. McCarthy, F. P. Glasser, D. M. Roy, and S. Diamond. Pittsburgh, PA: Materials Research Society, 1987. 99-108.

93. Sullentrup, Michael G. and Baldwin, J. W. "High Lime Fly Ash as a Cementing Agent." *ACI Special Publication* **79** (1983): 541-549.

94. Suzuki, J. and N. Tachibana. "Precipitability of Fly Ash From Various Coals." *Proceedings of the 2nd International Conference on Electrostatic Precipitators*, Nov.,1984, Koyoto, Japan. Editor Senichi Masuda. Pittsburgh, PA: Air Pollution Control Association, 1985. 194-199.

95. Swamy, R. N. *Cement Replacement Materials.* London: Blackie & Son, 1986.

96. Tamas, F. D. and K. Kovacs. "A Generalization and Computer Solution of the Bogue Calculation." *Cement Technology* **5** (1974): 393-401.

97. Thorne, D. J. and J. D. Watt. "Composition and Pozzolanic Properties of Pulverized Fuel Ashes II. Pozzolamic Properties of Fly Ashes as Determined by Crushing Strength Tests on Lime Mortars." *Journal of Applied Chemistry* **15** (December 1965): 595-604.

98. Van Hook, R. I. "Potential Health and Environmental Effects of Trace Elements and Radionuclides From Increased Coal Utilization." *Environmental Health Perspectives* **33** (1979): 227-247.

99. Watson, J. S. "A Survey of Ash Sources for Mineral Recovery." In *Fly Ash and Coal Conversion By-Products: Characterization, Utilization, and Disposal II*, Vol. 65. Eds. G. J. McCarthy, F. P. Glasser and D. M. Roy. Pittsburgh, PA: Materials Research Society, 1986. 59-67.

100. Watt, J. D. and D. J. Thorne. "Composition and Pozzolamic Properties of Pulverized Fuel Ashes I. Composition of Fly Ashes from Some British Power Stations and Properties of Their Component Particles." *Journal of Applied Chemistry* **15** (December 1965): 585-594.

101. Watt, J. D. and D. J. Thorne. "The Composition and Pozzolamic Properties of Pulverized Fuel Ashes III. Pozzolanic Properties of Fly Ashes as Determined by Chemical Methods." *Journal of Applied Chemistry* **16** (February 1966): 33-39.

102. Willard, H. H., L. L. Merritt, and J. A. Dean. *Instrumental Methods of Analysis.* 5th edition. New York, NY: D. Van Nostrand Company, 1974.

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APPENDIX A: REPEATABILITY TEST RESULTS

Repeatability Tests

One of the first tasks of this research was to verify the repeatability of the proposed testing methods for fly ash pastes. No other researchers had previously utilized fly ash pastes in their studies, and hence, little was known about the day-to-day testing repeatability that would be needed to monitor the physical properties of fly ash pastes. The purpose of this section is to report the results of three studies concerning the repeatability of the fly ash paste testing methods utilized throughout this dissertation. The first study simply addressed the precision of the compressive strength tests. Also, it was used to check the compressive strength data to see if they were normally distributed. The second study investigated the influence of water-fly ash ratio on the various physical properties of fly ash pastes. And finally, in the third phase of the study, the entire paste testing program (i.e., strength, time of set, volume stability and heat evolution properties) was subjected to a multi-day, multi-operator test to assess the repeatability that could be expected from the test results. Two fly ash samples were used in the first and third phases of the study. The two samples that were chosen were from Ottumwa generating station (OTT; sampling date 2/25/85), and Lansing generating station (LAN; sampling date 3/29/85). These two fly ash samples exhibited physical properties that were similar to many of the fly ash pastes that have been reported in this dissertation. Only the Lansing fly ash was used to study the influence of water:cement ratio on the various physical properties of fly ash pastes.

The first study began by making 60 compressive strength cubes from each sample of fly ash. The cubes from the respective fly ashes, were numbered from 1 to 60. Then a table of random numbers was used to separate the 60 cubes into 20 groups of three cubes each. The cubes were moist cured for seven days. After the curing period the cube specimens were broken in unconfined compression. The results of this phase of the study are listed in Tables A1 and A2. Table A1 lists the test statistics that were obtained when the cubes were treated on an individual

Table A1. Summary of cube strength repeatability tests (individual basis)

OTTUMWA PASTE CUBES

Mean:	Std. Dev.:	Std. Error:	Variance:	Coef. Var.:	Count:
1412.68333	170.686797	22.0355708	29133.9828	12.0824528	60
Minimum:	**Maximum:**	**Range:**	**Sum:**	**Sum of Sqr.:**	**# Missing:**
965	1834	869	84761	121459357	0

LANSING PASTE CUBES

Mean:	Std. Dev.:	Std. Error:	Variance:	Coef. Var.:	Count:
4160.98333	513.392852	66.2787321	263572.22	12.3382578	60
Minimum:	**Maximum:**	**Range:**	**Sum:**	**Sum of Sqr.:**	**# Missing:**
2667	5486	2819	249659	1054377699	0

Table A2. Summary of cube strength repeatability tests (grouped basis)

OTTUMWA PASTE CUBES

Mean:	Std. Dev.:	Std. Error:	Variance:	Coef. Var.:	Count:
1412.66	88.9656847	19.8933319	7914.89305	6.297742181	20
Minimum:	**Maximum:**	**Range:**	**Sum:**	**Sum of Sqr.:**	**# Missing:**
1264.7	1552.7	288	28253.2	40062548.48	0

LANSING PASTE CUBES

Mean:	Std. Dev.:	Std. Error:	Variance:	Coef. Var.:	Count:
4160.93	243.585695	54.4674172	59333.9906	5.854116619	20
Minimum:	**Maximum:**	**Range:**	**Sum:**	**Sum of Sqr.:**	**# Missing:**
3679	4533	854	83218.6	347394115	0

basis. Table A2 lists the test statistics that were obtained when the cubes were grouped into sets of three. Compressive strengths based on the average of three cube specimens were deemed as adequate for the purposes of this investigation (i.e., a coefficient of variation of less than 10%).

The following procedure was used to test the normality of the 60 repetitions of the compressive strength tests that were observed for each fly ash paste. The Statistical Analysis System (SAS) was used to perform the following calculations.

```
PROC RANK NORMAL = BLOM;
VAR LOAD;
RANK RANKITS;
PROC PLOT; PLOT LOAD*RANKITS;
PROC GLM; MODEL LOAD = RANKITS;
```

The program produces results similar to those obtained by plotting the data on probability paper. The straighter the line, then the better the data simulates a normal distribution. The results of the process are shown in Figures A1 and A2. For the purpose of this study the data were deemed to be normally distributed.

The Lansing fly ash was used to make paste mixtures with water:fly ash ratios of 0.27, 0.35, 0.45 and 0.55, respectively. The results of varying the water-fly ash ratio of pastes made with the Lansing fly ash are summarized in Table A3. In general, the results were similar to those observed for portland cement specimens because the decrease in compressive strength was inversely proportional to the water-fly ash ratio. This is in accordance with Abram's law, a limiting case of Feret's law, which is commonly applied to cement materials [46]. A plot of 7-day compressive strength versus water/fly ash ratio is shown in Figure A3. Similar results were obtained with specimens cured for other periods of time.

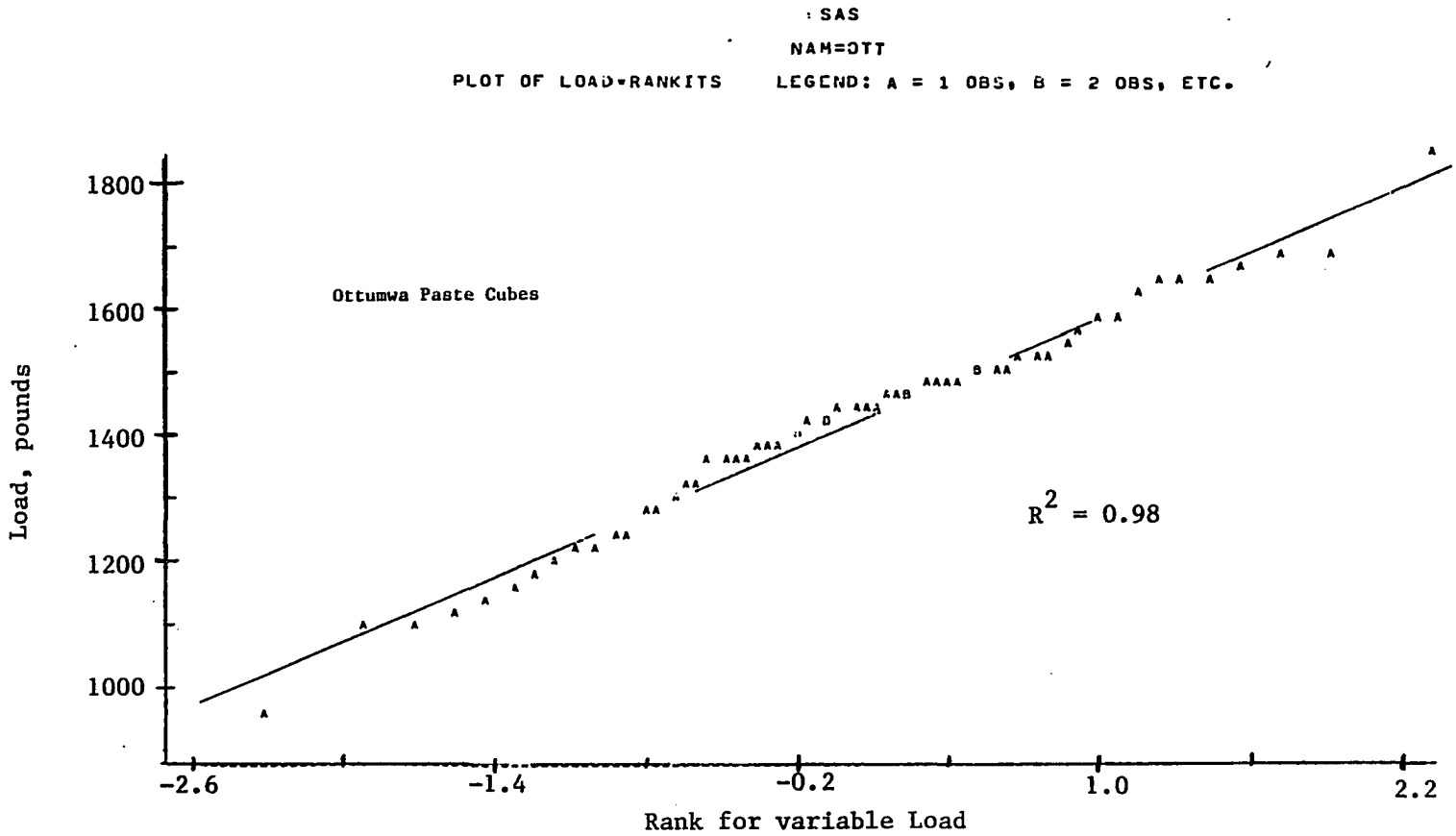

Figure A1. Normality test results for Ottumwa paste cubes

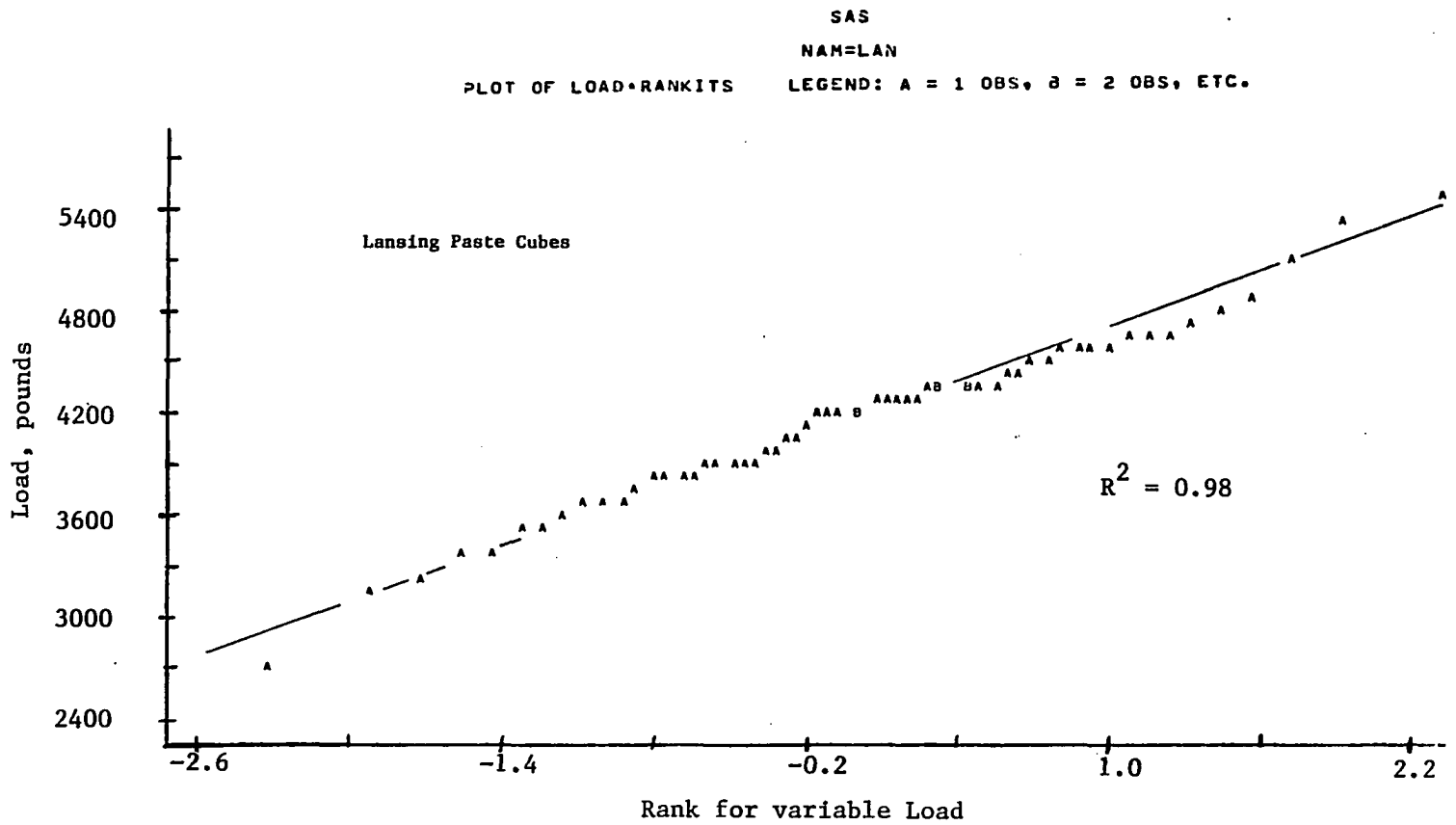

Figure A2. Normality test results for Lansing paste cubes

Table A3. Physical properties for Lansing fly ash pastes at different water-fly ash ratios

LANSING FLY ASH (sampled 3/29/85)

	Water/fly ash Ratio			
STRENGTH (PSI)	0.27	0.35	0.45	0.55
4-HOUR	2053	1041	659	429
1-DAY	3213	1607	1053	652
7-DAY	4610	3012	2082	1135
14-DAY	5613	3577	2444	1478
28-DAY	5080	4491	2857	1863
VOLUME STABILITY (% expansion, 28-days curing)				
Air Cured	-0.07	-0.11	-0.12	-0.16
Humid Cured	0.12	0.19	0.15	0.12
SET TIME (min.)				
Initial	10.0	8.5	10.0	11.0
Final	12.0	9.5	11.0	13.0

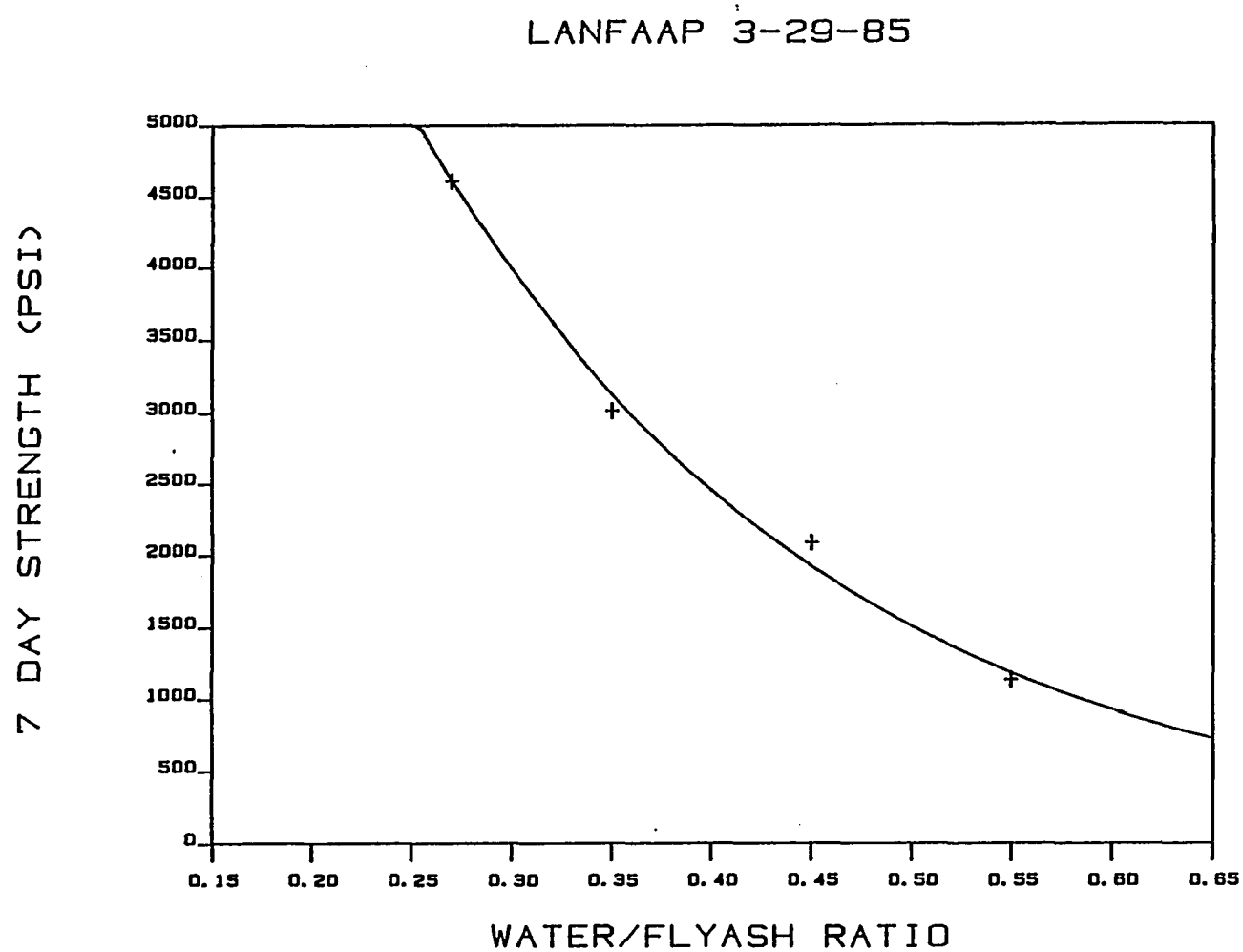

Figure A3. Influence of water content on compressive strength for Lansing paste cubes

Air cured expansion (i.e., drying shrinkage) of the paste specimens tended to increase with water/fly ash ratio. The results of the humid cured expansion test tended to decrease with increasing water/fly ash ratio.

Setting time of the fly ash paste specimens (both initial and final set) appeared to be independent of water-fly ash ratio for the range of values studied in this investigation (w/fa = 0.27 to 0.55). This may be important to the field utilization of fly ash grouts or slurries because it indicates that some type of retarder must be used to delay the flash setting characteristics of the mixtures. Increasing the water content will increase the fluidity of the mixture but it may not significantly alter the setting time for some fly ashes.

In general, the multi-day, multi-operator repeatability tests indicated that the methods used for characterizing the physical properties of the fly ash pastes were adequate (see Tables A4 and A5). Typically, the coefficients of variation for the compressive strength tests were about 10 to 20%. Hence, the tests are not precise enough to compare samples whose strengths differ by less than about 40%. It is pertinent to mention, however, that in this study, strength variations of greater than a factor of 5 (i.e., 500%) have been observed in a single power plant (OGS). Strength variations between power plants may also vary by a factor of 5 to 10. Thus, the tests were deemed adequate to study the trends in compressive strength of the fly ash pastes. Results of the remaining tests (i.e., volume stability, time of set and temperature rise) are also summarized in Tables A4 and A5. These tests were quite repeatable on a day-to-day basis.

Table A4. Repeatability tests on Lansing Fly Ash, sampled 3/29/85

	DAY 1		DAY 2		DAY 3		OVERALL	
	Mean	Std. Dev.	MEAN	Std. Dev.	MEAN	Std. Dev.	Mean	Std. Dev.
COMPRESSIVE STRENGTH (PSI)								
4-HOUR	—	—	2010	427	2096	195	2053	301
1-DAY	3171	132	3146	451	3321	230	3213	274
7-DAY	4915	850	4558	268	4356	427	4610	552
14-DAY	6039	807	5627	477	5172	370	5613	629
28-DAY	6134	—	4644	308	4680	—	5080	787
56-DAY	4499	1066	5822	816	5680	411	5334	943
VOLUME STABILITY (% exp. @ 28-days)								
Air Cured	-0.068	—	-0.062	—	-0.084	—	-0.071	0.011
Humid Cured	—	—	0.125	—	0.121	—	0.123	—
SET TIME (min.)								
Initial	9.5	—	10.0	—	10.5	—	10.0	0.5
Final	12.0	—	11.5	—	11.5	—	11.7	0.3
TEMPERATURE RISE								
DT (°C)	14.5	—	15.2	—	15.3	—	15.0	0.4
Peak Temp. (°C)	40.5	—	40.2	—	41.3	—	40.7	.6
Time to Peak(min)	23	—	22	—	20.5	—	21.8	1.3

Table A5. Repeatability tests on Ottumwa Fly Ash, sampled 2/25/85

	DAY 1		DAY 2		DAY 3		OVERALL	
	Mean	Std. Dev.	MEAN	Std. Dev.	MEAN	Std. Dev.	Mean	Std. Dev.
COMPRESSIVE STRENGTH (PSI)								
4-HOUR	601	138	574	78	635	46	603	87
1-DAY	752	43	814	82	629	24	744	92
7-DAY	993	36	1014	179	886	159	964	139
14-DAY	1264	—	1131	175	1009	150	1118	163
28-DAY	1079	121	1054	100	760	127	964	184
56-DAY	1038	—	1101	343	1168	191	1110	223
VOLUME STABILITY (% exp. @ 28-days)								
Air Cured	-0.035	—	-0.037	—	-0.046	—	-0.039	0.006
Humid Cured	0.002	—	-0.001	—	0.016	—	0.006	0.009
SET TIME (min.)								
Initial	16	—	18	—	18	—	17.3	1.2
Final	25	—	27	—	29	—	27	2.0
TEMPERATURE RISE								
DT (°C)	4.3	—	6.9	—	4.7	—	5.3	1.4
Peak Temp. (°C)	30.3	—	29.9	—	29.7	—	30.0	0.3
Time to Peak(min)	56	—	53	—	61	—	56.7	4.0

APPENDIX B: ASTM TESTING SUMMARY AND DATA

Description of Variables in Tables B1 and B2

Sample	=	source and sampling date of the fly ash
SiO2	=	concentration of Si, expressed as an oxide (wt. %, as received)
Al2O3	=	concentration of Al, expressed as an oxide (wt. %, as received)
Fe2O3	=	concentration of Fe, expressed as an oxide (wt. %, as received)
SO3	=	concentration of S, expressed as an oxide (wt. %, as received)
CaO	=	concentration of Ca, expressed as an oxide (wt. %, as received)
MgO	=	concentration of Mg, expressed as an oxide (wt. %, as received)
P2O5	=	concentration of P; expressed as an oxide (wt. %, as received)
K2O	=	concentration of K, expressed as an oxide (wt. %, as received)
Na2O	=	concentration of Na, expressed as an oxide (wt. %, as received)
TiO2	=	concentration of Ti, expressed as an oxide (wt. %, as received)
SrO	=	concentration of Sr, expressed as an oxide (wt. %, as received)
BaO	=	concentration of Ba, expressed as an oxide (wt. %, as received)
MC	=	moisture content (wt. %, as received)
LOI	=	loss on ignition at 750 deg. C (wt%, dry sample)
Avail Alk.	=	available alkalis (wt. %,equivilent sodium oxide)
Fineness	=	wt. % retained on a #325 mesh sieve
28D Pozz	=	strength index with portland cement at 28 days (% of control)
Water reqd	=	water requirement of mortar (% of control)
7D Pozz	=	strength index with portland cement at 7 days (% of control)
Autoclave	=	% expansion after 3 hours of autoclave curing
SG	=	specific gravity (dimensionless)
Day 1/1/83	=	days from Jan. 1, 1983, arbitrary reference baseline
blank or •	=	missing data, due to various reasons

Table B1. Raw data for ASTM chemical and physical testing

	A	B	C	D	E	F	G	H	I	J	K	L
1	Sample	SiO2	Al2O3	Fe2O3	SO3	CaO	MgO	P2O5	K2O	Na2O	TiO2	SrO
2	NE4020883	34.1	16.1	6.3	2.95	26.5	6.32	•	0.24	1.96	•	•
3	LAN021583	34.5	16.7	5.6	3.51	26.8	6.14	•	0.4	2.33	•	•
4	OTT021883	33.1	20.1	5.4	1.47	24.7	4.94	•	0.37	1.78	•	•
5	CBF031183	30.6	17.3	4.9	2.65	27.8	6.47	•	0.31	1.7	•	•
6	OTTCOMP1-83	35	20.2	5.3	1.72	25.2	5.13	1.63	0.39	1.69	1.46	•
7	CBF042883	31.8	16.7	5.3	3.29	28	6.82	1	0.34	1.76	1.32	•
8	OTTCOMP2-83	32.2	19.6	5.3	1.58	25.4	4.95	1.24	0.37	1.77	1.48	•
9	NE4060183	36.3	15.6	6.1	3.53	25.6	5.94	0.74	0.29	2.08	1.03	•
10	NE4060183	34.8	15.5	6.3	3.55	25.8	5.82	0.72	0.27	2.05	1.03	•
11	LAN062083	41.2	16.8	5.6	4.47	25.7	6.74	0.9	0.38	1.88	1.32	•
12	OTTCOMP3-83	34.3	20.2	5.3	1.76	25.1	4.99	1.86	0.39	1.91	1.48	•
13	CBF062483	32.2	16.9	5.3	3.03	27.2	6.58	0.72	0.29	1.89	1.4	•
14	LAN070183	33.7	17.5	5.2	2.84	27.2	7.3	1.33	0.36	2.04	1.26	•
15	OTTCOMP4-83	34.1	20.5	5.3	1.88	25.4	5.26	1.34	0.4	2	1.54	•
16	LOU080383	39.6	19.8	5.5	1.6	23.6	4.71	1.34	0.48	1.93	1.42	•
17	OTTCOMP5-83	37	20.3	5.1	1.57	24.2	4.86	1.32	0.45	1.92	1.48	•
18	OTTCOMP6-83	34.6	19.7	5	1.37	23.5	4.69	1.55	0.46	2.25	1.38	•
19	CBF082683	31.3	16.7	5.1	3.29	28.6	6.74	0.88	0.37	1.78	1.36	•
20	NE4090183	35.7	15.6	6.1	3.28	25.7	6.1	0.81	0.36	2.25	0.99	•
21	LAN091383	33.5	15.9	5.8	3.82	27.2	6.34	0.76	0.36	1.94	1.28	•
22	OTTCOMP7-83	35.5	19.3	5.2	2.02	24.3	4.71	0.94	0.41	2.36	1.5	•
23	OTTCOMP8-83	36.9	19.5	5.1	1.56	23.7	4.77	1.39	0.43	2.45	1.44	•
24	NE4012384	33.9	15.4	6.3	3.41	25.1	5.64	0.78	0.32	2.53	0.97	•
25	LOUCOMP1-84	34.3	18.8	5.4	1.23	23.8	4.11	1.16	0.42	1.82	1.44	•
26	LAN031584	35.1	15.8	5.3	3.36	26.9	6.49	0.92	0.44	1.94	1.25	•
27	OTTCOMP9-84	35.7	18.5	4.8	1.74	22.7	4.55	1.61	0.44	2.35	1.35	•
28	CBF033084	30.1	16.6	4.7	2.52	27.6	5.48	1.64	0.25	1.87	1.37	•
29	OTTCOMP10-84	33.7	17.9	5.1	1.92	24.5	4.56	1.66	0.36	2.53	1.42	•
30	LOUCOMP2-84	35.2	19.1	5.1	1.11	23.5	4.38	1.49	0.42	2.01	1.42	•
31	OTTCOMP11-84	35.1	18.3	5	1.72	23.9	4.9	2.21	0.37	2.38	1.33	•
32	NE4052384	33.9	15	5.6	3.33	26.8	5.97	0.87	0.29	2.37	0.99	•
33	OTTCOMP12-84	34.4	18.1	5	1.78	24	4.96	2.23	0.37	2.64	1.33	•
34	LOUCOMP3-84	37.5	18.7	5.2	1.31	24.2	4.88	1.98	0.36	1.8	1.39	•
35	CBF061484	34.2	17.4	4.9	2.7	27	5.56	1.14	0.38	1.97	1.42	•
36	CBFCOMP1-84	33.9	16.4	5.1	3.31	27.9	5.83	1.1	0.33	1.94	1.37	•
37	OTTCOMP13-84	34.1	18.3	5.3	2.11	24.4	4.6	1.86	0.37	2.83	1.42	•
38	LAN070384	31	15.4	6.3	4.16	27.7	6.19	0.86	0.27	1.9	1.17	•
39	OTTCOMP14-84	35.2	18.5	5.5	2.32	24.1	4.68	2.04	0.36	2.58	1.36	•
40	LOUCOMP4-84	36.2	18.8	5.6	1.44	24.8	4.84	1.87	0.36	1.99	1.45	•
41	NE4071884	34.7	16.6	5.9	3.21	25.7	6	1.29	0.3	2.38	1.13	•
42	OTTCOMP15-84	33.8	18.3	5.2	2.06	23.9	4.39	1.85	0.4	2.51	1.36	•
43	NE4072384	33	15.8	5.9	3.88	26.1	6.05	1.13	0.25	2.43	1.05	•
44	OTTCOMP16-84	34.5	18.5	5.3	2.34	23.6	4.43	1.65	0.4	2.34	1.39	•
45	OTTCOMP17-84	34.8	18.4	5.3	2.08	23.6	4.46	1.72	0.41	2.48	1.34	•
46	OTTCOMP18-84	38.2	18.6	5.1	2	22.3	4.42	1.73	0.46	2.78	1.28	•
47	OTTUMWA-84	35.5	18.4	5.3	2.27	24.1	4.75	1.99	0.37	2.89	1.39	•
48	OTTCOMP19-84	33.1	18	5.2	2.66	24.5	4.62	1.87	0.37	2.57	1.37	•
49	CBFCOMP2-84	34.2	18	5.2	2.54	26.7	5.69	1.14	0.38	1.86	1.38	•
50	OTTCOMP20-84	34.9	18	5.2	2.74	24.5	4.66	1.79	0.38	2.73	1.43	•
51	LANCOMP1-84	32.8	15.6	5.6	4.78	27.4	5.96	0.86	0.35	1.84	1.2	•
52	CBFCOMP3-84	35.3	17.3	5.1	2.79	25.7	5.6	1.27	0.36	2.07	1.28	•
53	NE4COMP1-84	31.9	15.5	5.7	4.51	25.9	5.53	0.87	0.29	2.8	1.06	•
54	CBFCOMP4-84	33.5	17	5.1	2.5	25.7	5.46	1.23	0.35	1.85	1.2	•
55	OTTCOMP21-84	36.8	18.4	5.2	2.88	22.3	4.58	1.48	0.48	2.6	1.32	•
56	NE4COMP2-84	34.3	15.8	5.6	4.57	25.6	5.69	0.87	0.33	2.74	1.07	•
57	CBFCOMP5-84	34.4	17.4	5.4	3.04	27.2	6.06	1.19	0.34	2.29	1.31	•
58	OTTCOMP22-84	36.9	18.1	5.4	2.14	23.8	4.61	1.6	0.43	2.57	1.4	•
59	LANCOMP2-84	38.4	15.5	5.6	4.88	25.3	5.62	0.73	0.54	1.84	1.17	•
60	OTTCOMP23-84	37.1	18.4	5.3	2.45	24.3	4.73	1.84	0.42	2.67	1.44	•
61	OTTCOMP1-85	31.7	18.1	4.8	2.57	24.4	4.89	1.81	0.41	2.73	1.39	•
62	NE4022585	35.2	15.4	5.8	4.24	26.5	5.99	0.74	0.25	2.49	1.04	•
63	OTT022585	34.6	18.5	4.8	3.68	24.9	5.28	1.11	0.42	3.28	1.43	•
64	NE4031985	31.1	14.9	5.6	4.56	26.8	5.62	0.77	0.24	2.23	1.04	•
65	LAN032985	31.1	15.9	5.3	3.94	28.1	5.72	1.25	0.27	1.62	1.35	•

	M	N	O	P	Q	R	S	T	U	V	W
1	BaO	MC	LOI	Avail Alk.	Fineness	28D Pozz	Water reqd	7D Pozz	Autoclave	SG	Day 1/1/83
2	•	0.04	0.16	1.39	8.2	100	88	•	0.09	2.69	39
3	•	0.01	0.22	1.58	11.5	86	88	•	0.11	2.72	46
4	•	0.06	0.19	1.15	9.9	108	88	•	0.06	2.62	49
5	•	0.06	0.19	1.18	15.3	94	86	•	0.12	2.69	70
6	•	0.03	0.31	1.31	9.9	106	86	•	0.06	2.63	90
7	•	0.08	0.48	1.31	10.5	101	88	•	0.14	2.75	118
8	•	0.05	0.19	1.32	10	109	98	•	0.07	2.84	146
9	•	0.01	0.19	1.45	6	113	88	•	0.09	2.69	152
10	•	0.02	0.16	1.57	4.9	110	88	•	0.07	2.66	152
11	•	0.05	0.2	1.33	12.8	89	95	•	0.09	2.81	171
12	•	0.03	0.28	1.36	9.8	82	86	•	0.06	2.64	173
13	•	0.05	0.36	1.4	8.5	103	96	•	0.13	2.72	175
14	•	0.02	0.76	1.41	16.2	75	100	•	•	2.75	182
15	•	0.03	0.25	1.45	10.7	92	87	•	0.06	2.62	204
16	•	0.04	0.3	1.42	6.7	93	86	•	0.06	2.69	215
17	•	0.04	0.28	1.36	10.5	112	89	•	0.06	2.58	226
18	•	0.07	0.17	1.6	10.1	105	89	•	0.05	2.6	236
19	•	0.08	0.27	1.35	8.7	97	96	•	0.15	2.69	238
20	•	0.02	0.17	1.43	10.7	94	89	•	0.07	2.71	244
21	•	0.07	0.56	1.37	11	93	99	•	0.13	2.8	256
22	•	0.02	0.27	1.74	10.2	111	92	•	0.01	2.59	279
23	•	0.03	0.25	1.7	10.6	103	92	•	0.01	2.58	345
24	•	0.01	0.34	1.73	11.7	97	100	•	0.03	2.63	396
25	•	0.02	0.16	1.29	8	98	92	90	0.01	2.63	426
26	•	0.02	0.33	1.46	15.2	93	90	93	0.06	2.78	439
27	•	0.06	0.26	1.25	11.1	89	90	99	0	2.55	443
28	•	0.02	0.38	1.3	11.1	106	90	99	0.08	2.71	464
29	•	0.02	0.25	1.77	9.6	104	90	94	0.04	2.62	491
30	•	0.03	0.21	1.15	7	104	90	99	0.02	2.55	506
31	•	0.03	0.17	1.46	9.4	98	91	90	0.03	2.61	507
32	•	0.01	0.23	1.4	10.5	83	91	92	0.05	2.74	509
33	•	0.03	0.2	1.73	9.9	93	90	97	0.03	2.61	524
34	•	0.02	0.15	1.02	6.6	68	90	92	0.03	2.61	530
35	•	0.07	0.31	1.03	7.7	117	90	64	0.06	2.62	534
36	•	0.04	0.53	1.19	11.6	93	90	80	0.09	2.68	539
37	•	0.05	0.26	1.87	8.8	95	90	91	0.03	2.66	540
38	•	0.01	0.26	1.26	10	99	90	91	0.08	2.81	549
39	•	0.02	0.3	1.72	10.3	95	90	96	0.03	2.64	555
40	•	0.24	0.34	0.9	7.4	102	90	94	0.03	2.62	559
41	•	0.01	0.27	1.51	12.5	95	90	95	0.05	2.64	566
42	•	0.05	0.27	1.08	10.5	102	90	87	0.03	2.57	569
43	•	0.08	0.36	1.45	11.1	89	90	79	0.06	2.64	572
44	•	0.04	0.24	1.03	11.2	97	90	95	0.03	2.54	578
45	•	0.01	0.36	1.15	11	107	90	94	0.04	2.6	588
46	•	0.02	0.32	1.1	10.5	105	90	98	0.03	2.59	595
47	•	0.01	0.3	1.82	9.9	94	90	85	0.04	2.61	600
48	•	0.01	0.22	1.79	9.9	87	89	85	0.04	2.61	603
49	•	0.09	0.51	1.31	12.2	98	90	88	0.07	2.71	604
50	•	0.02	0.24	1.72	10.4	98	90	86	0.04	2.6	612
51	•	0.08	0.34	1.38	9.1	93	90	89	0.08	2.78	813
52	•	0.09	0.46	1.34	12.4	95	90	91	0.05	2.6	617
53	•	0.04	0.37	1.79	11.6	91	90	89	0.08	2.65	625
54	•	0.05	0.46	1.25	12.2	96	90	95	0.07	2.6	631
55	•	0.04	0.28	1.48	11.5	96	90	87	0.04	2.54	633
56	•	0.03	0.3	1.54	11.5	87	90	87	0.07	2.63	639
57	•	0.08	0.49	1.55	14.4	101	92	91	0.07	2.64	644
58	•	0.03	0.24	1.8	11.2	104	90	84	0.04	2.58	658
59	•	0.06	0.23	1.22	9.9	80	90	88	0.06	2.76	658
60	•	0.03	0.21	1.73	9.9	95	92	76	0.03	2.61	687
61	•	0	0.23	2.05	11.3	88	92	91	0.06	2.61	784
62	•	0.01	0.33	1.54	12.9	99	86	88	0.07	2.66	787
63	•	0.01	0.38	1.97	11.3	100	83	87	0.06	2.58	787
64	•	0	0.24	1.56	15	94	90	86	0.11	2.69	799
65	•	0	0.35	1.13	12.8	89	91	90	0.11	2.79	822

Table B1. (Continued)

	A	B	C	D	E	F	G	H	I	J	K	L
66	CBFCOMP1-85	29.3	15.1	5.9	4.2	28.5	6.2	1	0.3	2	1	•
67	OTTCOMP2-85	31.6	17.7	4.9	3.18	24.3	4.78	1.81	0.4	3.13	1.35	•
68	CBF042585	28.6	16.3	5.1	4.19	28.6	5.07	1.01	0.3	1.81	1.38	•
69	LAN050185	30.3	14.7	6.5	4.6	28.2	5.94	0.85	0.27	2.12	1.02	•
70	OTTCOMP3-85	32.4	18	5.2	2.14	24	4.74	1.94	0.42	2.08	1.36	•
71	NE4COMP1-85	34.4	15.7	5.7	3.5	26.2	8.21	0.93	0.28	2.29	1.03	•
72	OTTCOMP4-85	32.6	18.1	6.1	2.34	25.7	4.84	1.7	0.34	2.04	1.47	•
73	CBF051085	29	15.8	5.2	4.37	28.1	5.54	1.1	0.3	1.91	1.19	•
74	OTTCOMP5-85	30.8	18	5.9	2.3	26	5.02	2.13	0.33	1.83	1.43	•
75	NE4COMP2-85	35.8	15.8	5.9	3.33	25.5	5.91	0.95	0.32	2.28	1.03	•
76	OTTCOMP6-85	32.3	18.1	5.9	2.27	25.4	4.69	2.18	0.36	1.87	1.39	•
77	CBFCOMP2-85	32.6	16.4	5	3.08	27.7	5.55	1.37	0.32	1.83	1.33	•
78	LANCOMP1-85	29.2	15.6	6	4.5	28.1	5.69	0.87	0.27	2.06	1.2	•
79	OTTCOMP7-85	38	18.7	5.7	2.34	24.7	5.02	2	0.39	1.82	1.38	•
80	LAN061985	32.8	15.6	6.2	4.37	27.9	6.04	0.87	0.26	2.26	1.22	•
81	NE4COMP3-85	35.5	16.1	5.4	3.33	25.6	5.75	1	0.33	2.17	1.02	•
82	NE4062185	36.6	15.9	5.9	3.53	25.6	6.04	0.99	0.32	2.4	1.02	•
83	OTTCOMP8-85	32.6	18.8	5.1	2.47	25	4.93	1.33	0.4	1.52	1.41	•
84	CBF000085	31	15.1	6.2	4.08	28.5	6.52	0.94	0.21	2.27	1.06	•
85	OTTCOMP9-85	33.3	19	5.3	2.94	25.1	4.87	1.14	0.41	2.01	1.46	•
86	NE4COMP4-85	32.4	16.3	5.6	3.25	26.4	5.68	1.02	0.32	2.01	1.04	•
87	CBFCOMP3-85	31.8	15.3	5.7	4.01	28	6.04	0.91	0.27	2.03	1.19	•
88	OTTCOMP10-85	30.8	18.5	5.7	2.75	25.4	4.85	1.44	0.39	2.13	1.44	•
89	NE4COMP5-85	34.3	15.8	6.1	3.41	26.2	5.74	0.88	0.3	1.91	1.04	•
90	OTTCOMP11-85	34.4	18.7	5.4	2.69	25.1	4.94	1.4	0.42	2.15	1.48	•
91	LANCOMP2-85	31.7	15.2	6	4.39	27.8	6.02	0.78	0.27	2.33	1.13	•
92	OTTCOMP12-85	31.3	18.5	5.7	2.33	25.2	4.85	1.94	0.39	2.41	1.4	•
93	NE4COMP6-85	33.6	15.8	6.1	3.64	26.8	5.76	0.93	0.27	2.31	1.04	•
94	NE4COMP7-85	33.3	15.8	6.2	3.76	27.2	5.8	0.85	0.25	2.1	1.03	•
95	CBFCOMP4-85	33.1	15.8	5.2	3.2	27.5	5.9	0.89	0.29	1.8	1.31	•
96	OTTCOMP13-85	30.9	18	5.8	3	25.6	4.8	1.24	0.35	2.38	1.44	•
97	NE4COMP8-85	33.9	16	6	3.2	26.3	5.9	0.81	0.3	1.78	1.01	•
98	LANCOMP3-85	33.2	15.6	5.8	3.8	26.9	5.8	0.82	0.36	1.92	1.21	•
99	CBFCOMP5-85	30.1	15.7	5.4	3.7	28.5	6.2	0.72	0.23	1.78	1.3	•
100	NE4091785	37.8	18	4.7	2	22.8	5	1.48	0.42	2.56	1.22	•
101	NE4COMP9-85	35	16.7	5.5	2.8	25.3	5.6	1.03	0.34	1.89	1.05	•
102	OTTCOMP14-85	30.7	18.2	5.5	2.4	25.3	4.91	1.36	0.36	1.91	1.4	•
103	NE4092485	41.1	17.8	4.5	2.1	22.3	4.9	1.2	0.46	2.38	1.21	•
104	NE4COMP10-85	38.5	17.7	5	2.09	22.4	4.87	1.21	0.46	2.26	1.1	•
105	LANCOMP4-85	32.2	16.1	5.8	4.83	26.6	5.15	0.61	0.31	2.14	1.25	•
106	OTTCOMP15-85	32.4	18.4	5.4	2.27	25.6	5.04	0.98	0.37	1.33	1.45	•
107	CBFCOMP6-85	31.8	16.9	4.8	3.22	27.7	5.19	1.05	0.32	1.79	1.41	•
108	OTTCOMP16-85	29.5	18.4	5.3	1.92	25.4	4.97	1.53	0.37	1.66	1.41	•
109	LANSING-86	31.5	16.1	6.7	3.79	29.1	5.51	0.77	0.23	1.91		0.39
110	NE4021286	33.2	18	5.7	2.37	25.4	4.87	2.08	0.34	2.28		0.54
111	CBF021986	31.4	16.6	5.4	3.49	32.1	5.59	1.45	0.18	1.53		0.45
112	NE4(Caballo)-86	32.8	18.1	5.7	2.36	26.1	4.71	2.09	0.33	2.29		0.54
113	LAN030786	31.6	16	8.3	3.95	30.3	5.77	0.8	0.26	1.95		0.39
114	OTT031386	29.6	19	6	2.45	27.3	4.55	1.62	0.38	2.13		0.46
115	CBF040486	30.4	17.4	5.1	3.1	32.4	5.33	1.71	0.24	1.61		0.49
116	OTTCOMP1-86	29.7	18.7	6.2	2.74	25.3	4.47	1.44	0.36	2.45		0.45
117	LANCOMP1-86	30.1	16.4	5.8	3.57	28.9	6.51	0.92	0.3	1.73		0.44
118	CBFCOMP1-86	30.1	16.9	5.5	3.21	29.5	4.9	1.3	0.26	1.76		0.42
119	OTTCOMP2-86	32	19.3	6.1	2.28	24.9	4.74	1.54	0.4	1.56		0.46
120	NE4051286	32.4	15.8	6.3	3.31	27	5.43	0.88	0.22	2.34		0.41
121	NE4COMP1-86	31.7	16	6	3.37	27.7	5.81	0.87	0.23	2.4		0.42
122	OTTCOMP3-86	33.8	19.2	6.2	1.86	25.1	4.56	1.53	0.41	1.51		0.45
123	LAN052086	29.2	16.1	5.9	3.97	30.3	6.71	0.81	0.2	2.07		0.4
124	CBFCOMP2-86	31.1	17.1	5.5	2.86	29.5	5.13	1.42	0.28	1.8		0.43
125	NE4COMP2-86	31.2	15.8	8	3.48	28.1	6.09	0.81	0.24	2.19		0.42
126	OTTCOMP4-86	30.8	18.5	6.2	2.3	25.4	4.4	2.08	0.39	1.97		0.51
127	NE4COMP3-86	31.5	15.7	6.1	3.32	27.9	5.65	0.72	0.27	2.08		0.46
128	OTTCOMP5-86	30	18.8	6.2	2.68	26.4	4.76	1.38	0.33	1.89		0.44
129	NE4COMP4-86	31.5	15.6	5.9	3.23	27.3	5.43	0.78	0.28	2.13		0.41
130	CBFCOMP3-86	30.5	16.8	5.4	3.04	30.3	5.24	1.24	0.27	1.71		0.42

	M	N	O	P	Q	R	S	T	U	V	W
66	•	0.01	0.56	1.34	19.1	84	92	80	0.14	2.7	823
67	•	0.02	0.29	2.16	10.6	82	89	92	0.08	2.63	836
68	•	0.04	0.4	1.2	14.9	88	89	90	0.12	2.88	850
69	•	0.03	0.21	1.27	16.8	79	89	93	0.12	2.8	852
70	•	0.02	0.28	1.63	10.4	99	86	92	0.03	2.68	852
71	•	0.02	0.27	1.55	15.9	96	88	93	0.09	2.7	861
72	•	0.01	0.17	1.55	10.2	88	89	91	0.08	2.68	865
73	•	0.12	0.48	1.34	16.2	86	87	83	0.09	2.74	866
74	•	0.02	0.17	1.5	10.1	94	86	97	0.07	2.69	879
75	•	0.05	0.24	1.57	14.1	95	88	90	0.08	2.62	879
76	•	0.04	0.21	1.48	9.2	88	86	96	0.07	2.68	886
77	•	0.21	0.76	1.45	14.2	85	88	82	0.11	2.7	894
78	•	0.07	0.47	1.84	12.4	83	89	83	0.12	2.8	895
79	•	0.05	0.19	1.43	10.4	93	86	91	0.06	2.64	896
80	•	0.02	0.77	1.7	12.4	85	88	91	0.12	2.82	901
81	•	0.06	0.38	1.73	10.7	97	88	84	0.09	2.58	901
82	•	0.03	0.32	1.68	10.1	84	90	89	0.09	2.58	903
83	•	0.02	0.25	1.29	9.5	97	86	89	0.07	2.63	906
84	•	0.02	0.34	1.62	10.8	85	88	83	0.15	2.76	914
85	•	0.03	0.33	1.59	9.9	89	86	90	0.05	2.62	922
86	•	0.04	0.3	1.48	12.1	97	88	93	0.06	2.57	932
87	•	0.15	0.61	1.49	12.8	94	88	92	0.11	2.72	935
88	•	0.02	0.21	1.6	9.1	90	86	93	0.06	2.68	940
89	•	0.06	0.27	1.34	10.7	88	88	104	0.07	2.62	949
90	•	0.04	0.24	1.42	9.8	105	86	96	0.05	2.62	950
91	•	0.04	0.54	1.62	11.2	89	89	99	0.11	2.79	950
92	•	0.03	0.32	1.65	8.8	102	88	98	0.04	2.66	959
93	•	0.06	0.35	1.61	9.2	82	88	91	0.07	2.65	962
94	•	0.03	0.32	1.46	9.2	93	89	97	0.07	2.64	970
95	•	0.07	0.37	1.26	12.1	92	89	90	0.1	2.7	971
96	•	0.06	0.26	1.65	9.6	90	88	89	0.05	2.66	977
97	•	0.06	0.38	1.17	11.5	92	89	93	0.07	2.62	982
98	•	0.14	0.56	1.35	11	95	90	87	0.09	2.77	988
99	•	0.1	0.37	1.2	10.4	97	89	89	0.1	2.72	998
100	•	0.02	0.3	1.05	8.8	107	89	98	0.06	2.53	991
101	•	0.02	0.3	1.09	9.7	102	89	94	0.06	2.57	991
102	•	0.02	0.31	1.27	9.9	97	86	82	0.04	2.66	995
103	•	0.02	0.28	1.12	11.6	102	89	95	0.04	2.42	998
104	•	0.03	0.31	0.84	9.8	101	89	96	0.04	2.45	1007
105	•	0.06	0.41	1.36	12.8	88	90	87	0.04	2.75	1008
106	•	0.04	0.25	0.84	10.2	98	87	96	0.04	2.64	1012
107	•	0.09	0.35	1.21	9.2	92	89	91	0.07	2.66	1015
108	•	0.01	0.2	1.1	9.6	101	87	92	0.05	2.65	1026
109	0.64	0.04	0.24	1.55	9.1	90	88	86	0.118	2.82	1128
110	0.86	0.05	0.29	1.57	12.1	96	89	91	0.067	2.6	1139
111	0.66	0.01	0.24	1.1	10.3	84	88	91	0.153	2.76	1146
112	0.86	0.04	0.31	1.74	11.8	96	89	90	0.074	2.63	1159
113	0.62	0.07	0.45	1.41	10.2	91	90	85	0.114	2.79	1168
114	0.72	0.06	0.2	1.57	9.3	109	87	101	0.079	2.64	1168
115	0.7	0.08	0.42	1.25	10.6	99	89	99	0.119	2.69	1195
116	0.69	0.08	0.23	1.36	8.3	103	87	98	0.073	2.69	1202
117	0.61	0.02	0.71	1.36	14.8	98	89	89	0.139	2.78	1202
118	0.67	0.04	0.41	1.37	9.2	96	89	92	0.12	2.71	1220
119	0.7	0.03	0.31	1.1	9.1	105	88	92	0.07	2.69	1225
120	0.73	0.04	0.23	1.58	10.5	101	88	84	0.091	2.69	1228
121	0.72	0.03	0.26	1.84	11	92	90	89	0.11	2.7	1239
122	0.71	0.02	0.27	1.13	8.7	103	88	96	0.065	2.72	1244
123	0.62	0.04	0.75	1.54	13.7	88	90	92	0.174	2.8	1249
124	0.69	0.1	0.32	1.33	8.9	97	88	95	0.117	2.7	1253
125	0.72	0.04	0.42	1.62	11.5	87	90	84	0.109	2.72	1254
126	0.8	0.05	0.39	1.44	8.7	100	88	88	0.001	2.7	1260
127	0.96	0.03	0.35	1.6	12.4	84	90	90	0.028	2.72	1268
128	0.66	0.03	0.31	1.31	9.1	90	88	92	-0.007	2.7	1275
129	0.7	0.06	0.5	1.51	12.1	92	90	87	0.028	2.68	1278
130	0.66	0.06	0.52	1.18	11.2	91	90	90	0.051	2.71	1286

Table B1. (Continued)

	A	B	C	D	E	F	G	H	I	J	K	L
131	NE4COMP5-86	30	15.6	6.3	3.56	27.8	5.87	0.84	0.22	2.38		0.41
132	OTTCOMP6-86	29.8	18.5	6.4	2.72	26.3	4.58	1.4	0.32	2.31		0.44
133	LANCOMP2-86	29.9	16.1	5.8	3.57	29.9	6.15	1.09	0.24	1.8		0.43
134	NE4COMP6-86	30.9	15.7	6.3	3.44	26.8	5.71	0.85	0.21	2.31		0.44
135	CBFCOMP4-86	29.2	16.6	5.1	3.46	31.7	5.09	1.04	0.23	1.46		0.43
136	OTTCOMP7-86	31.7	18.5	6	2.37	25.4	4.63	1.85	0.38	1.94		0.49
137	OTTCOMP8-86	32	18.5	5.7	2.31	24.7	4.55	1.58	0.39	1.94		0.47
138	LANCOMP3-86	31.1	16.5	5.8	3.56	29.6	5.46	0.98	0.28	1.72		0.41
139	OTTCOMP9-86	30.9	18.8	5.9	2.51	25.1	4.57	1.53	0.36	2.06		0.45
140	CBFCOMP5-86	30.3	16.7	5.3	3.26	30.4	4.9	1.23	0.27	1.45		0.51
141	OTTCOMP10-86	31.7	18.1	5.9	2.52	25.3	4.47	1.83	0.35	2.12		0.49
142	OTTCOMP11-86	30.7	18	5.8	2.83	25.5	4.81	1.77	0.33	2.24		0.48
143	LANCOMP4-86	30.2	16.8	5.6	3.46	30.4	5.36	1.01	0.24	1.14		0.43
144	OTTCOMP12-86	29.8	18.4	5.8	2.87	26.1	5.24	1.65	0.3	2.09		0.47
145	OTTCOMP13-86	30.6	18.4	5.7	2.64	25.2	4.86	1.64	0.34	2.12		0.46
146	LANCOMP5-86	29.7	16.8	5.6	3.76	30.4	5.55	1.01	0.26	1.39		0.42
147	OTTCOMP14-86	31	18.3	5.6	2.88	25.3	4.95	1.77	0.34	1.9		0.48
148	OTTCOMP15-86	31.4	18.8	5.9	2.49	26.5	4.87	1.84	0.35	1.88		0.48
149	CBF010987	27.6	17.3	5.6	3.17	31	6.43	1.28	0.22	1.48		0.47
150	CBF030487	29.2	17	7.1	3.52	28.3	6.68	0.61	0.24	2.02		0.41
151	LAN032887	32.2	16.7	5.9	3.5	28.7	5.43	1.04	0.24	1.68		0.44
152	OTTCOMP1-87	31	18.2	5.7	2.83	25	4.6	1.55	0.33	2.96		0.54
153	OTTCOMP2-87	30.6	18	5.8	3.34	25.5	4.65	1.47	0.32	3.21		0.44
154	LANCOMP1-87	29.9	16.9	6.3	3.66	29.3	7.12	0.72	0.24	2.04		0.4
155	OTTCOMP3-87	32.1	18.4	5.7	2.61	25.7	4.55	1.69	0.35	2.61		0.47
156	NE4050787	39.3	15.7	5.6	3.33	22.1	4.35	1.12	0.64	2.84		0.41
157	NE4COMP1-87	35.6	17.6	5.5	2.18	24	4.62	1.34	0.38	2.38		0.45
158	LANCOMP2-87	31.3	15.7	6.6	4.09	28.7	6.5	0.74	0.21	2.09		0.42
159	NE4COMP2-87	35.2	18	5.5	1.89	23.4	4.57	1.28	0.39	2.13		0.44
160	CBF060887	34.9	17	6.3	2.22	26.8	5.57	0.9	0.38	1.45		0.41
161	NE4COMP3-87	36.6	18	5.5	1.92	23.9	4.54	1.21	0.38	2.01		0.43
162	OTTCOMP4-87	37.6	17.3	5.3	1.72	23.8	4.39	1.53	0.45	1.6		0.46
163	LAN061987	31.3	16.9	5.7	3.73	28	5.13	1.32	0.22	1.65		0.53
164	NE4COMP4-87	36.3	17.8	5.6	1.84	23.7	4.46	1.13	0.38	1.98		0.42
165	OTTCOMP5-87	33.5	18.5	5.5	2.12	25.4	4.64	1.38	0.39	1.89		0.49
166	CBFCOMP1-87	31	16.2	6.4	2.96	28.6	5.99	0.8	0.25	1.63		0.42
167	NE4COMP5-87	35.6	17.7	5.7	2.01	24.2	4.6	1.15	0.36	2.1		0.42
168	OTTCOMP6-87	32.1	18.2	5.5	2.44	26.3	4.54	1.49	0.32	1.82		0.46
169	LANCOMP3-87	31.6	17.6	5.9	3.3	28.3	5.15	1.23	0.29	1.65		0.45
170	OTTCOMP7-87	30.2	17.9	5.9	3.1	26.6	4.33	1.39	0.29	2.71		0.43
171	NE4COMP6-87	35.6	17.7	5.6	1.99	23.9	4.75	1.09	0.39	2.16		0.41
172	OTTCOMP8-87	31.4	18	5.8	2.76	26.3	4.79	2.08	0.32	2.52		0.52
173	LANCOMP4-87	30.1	17	5.7	3.09	27.8	5.79	0.97	0.26	1.69		0.5
174	CBFCOMP2-87	30	16	6.5	3.2	29	6.74	0.74	0.22	1.84		0.41
175	OTTCOMP9-87	31.3	18.1	5.9	2.6	25.3	4.49	2.31	0.36	2.72		0.54
176	NE4COMP7-87	36.4	18.2	5.6	1.85	23.4	4.43	1.19	0.45	2.16		0.43
177	OTTCOMP10-87	30	18.3	5.2	3	24.8	4.4	1.48	0.32	2.88		0.48
178	NE4COMP8-87	35.5	17.5	5.5	2.12	23	4.63	1.12	0.42	2.15		0.41
179	OTTCOMP11-87	32.9	19.4	5.4	3.28	27.3	4.95	1.15	0.34	3.26	1.56	0.39
180	OTTCOMP12-87	33.6	19.2	5.4	3.04	26.6	4.75	1.25	0.36	2.79	1.52	0.39
181	CBFCOMP3-87	30.6	15.8	6.6	3.26	29	6.75	0.73	0.22	1.93		0.41
182	OTTCOMP13-87	30.8	17.7	5.6	3.58	26.2	4.48	0.9	0.27	3.22		0.4
183	NE4COMP9-87	35.5	17.7	5.5	2	23.2	4.5	1.1	0.39	1.97		0.41
184	CBFCOMP4-87	30.3	16.1	6.4	3.26	28.2	6.47	0.62	0.2	1.85		0.4
185	CBFCOMP5-87	29.7	16.4	6.7	3.42	28.9	6.52	0.6	0.21	1.78		0.39
186	OTTCOMP14-87	33.7	19.3	5.6	1.86	23.7	4.28	1.66	0.43	2.03		0.49
187	NE4COMP10-87	33.4	18.5	5.6	2.18	24	4.63	0.99	0.35	2.06		0.41
188	CBFCOMP6-87	29.3	16.4	7	3.49	29.4	6.66	0.61	0.24	1.83		0.4
189	OTTCOMP15-87	33.8	19.4	5.7	1.99	24.9	4.58	1.55	0.44	2.08		0.47
190	LAN102387	30.6	16.7	6.7	4.01	29	6.14	0.75	0.22	2.14		0.41
191	NE4103087	34.6	18.2	5.5	2.11	24.5	4.4	1.17	0.37	2.67		0.42
192	OTTCOMP16-87	34.6	19.5	5.6	1.79	23.7	4.35	1.59	0.46	1.98		0.49
193	OTT121587	33.6	19.6	5.5	2.09	24	4.3	1.26	0.42	2.29		0.44
194	LAN121387	29.3	17.1	6.3	4.12	29.9	5.68	0.92	0.24	1.91		0.42
195	CBF122287	29	16.5	7.1	3.61	29.1	6.51	0.68	0.2	1.91		0.41

	M	N	O	P	Q	R	S	T	U	V	W
131	0.74	0.06	0.33	1.5	12.2	87	89	85	0.031	2.69	1289
132	0.66	0.08	0.42	1.51	9.5	89	90	89	0.012	2.7	1293
133	0.67	0.06	0.72	1.29	13	103	91	90	0.08	2.79	1294
134	0.68	0.06	0.43	1.64	12.7	82	93	82	0.019	2.67	1298
135	0.62	0.1	0.46	1.07	10.6	84	95	86	0.079	2.72	1307
136	0.74	0.05	0.29	0.98	10.1	90	89	92	0.004	2.68	1309
137	0.67	0.04	0.22	1.15	9.8	96	91	100	0.004	2.66	1323
138	0.66	0.03	0.36	1.31	10.1	84	94	96	0.044	2.78	1328
139	0.69	0.03	0.21	1.19	9.7	100	92	93	-0.001	2.68	1332
140	0.63	0.18	0.63	1.62	9	84	92	90	0.055	2.7	1337
141	0.74	0.02	0.25	1.48	9.6	97	88	102	0.003	2.68	1341
142	0.72	0.09	0.4	1.47	9.6	100	90	93	0.011	2.69	1351
143	0.61	0.08	0.6	0.88	10.5	94	94	91	0.065	2.78	1358
144	0.68	0.04	0.28	1.57	10.1	103	89	96	0.015	2.69	1359
145	0.68	0.01	0.36	1.44	11.3	95	90	93	0.012	2.68	1371
146	0.64	0.03	0.38	1.09	10.7	95	94	87	0.058	2.79	1384
147	0.72	0.03	0.35	1.17	10	100	90	89	0.006	2.67	1384
148	0.73	0.02	0.3	1.04	9.9	92	91	81	0.016	2.68	1419
149	0.69	0.01	0.25	1.13	12.3	92	91	84	0.08	2.72	1474
150	0.6	0	0.26	1.49	12.6	84	92	81	0.12	2.71	1531
151	0.64	0	0.61	1.27	15.1	95	95	88	0.11	2.75	1552
152	0.68	0.04	0.41	2.5	11.4	91	91	83	0.092	2.66	1561
153	0.66	0.02	0.35	2.61	10.1	90	91	86	0.093	2.64	1576
154	0.61	0.02	0.74	1.53	14.3	90	95	84	0.141	2.77	1578
155	0.7	0.08	0.29	2.12	11.2	98	91	84	0.08	2.65	1588
156	0.62	0.02	0.27	1.81	12	84	91	82	0.082	2.58	1589
157	0.69	0.04	0.3	1.82	15.5	102	92	92	0.074	2.55	1599
158	0.61	0.03	0.35	1.64	9	94	92	94	0.124	2.78	1602
159	0.67	0.03	0.25	1.74	11.9	105	94	96	0.062	2.54	1612
160	0.6	0.01	0.19	1.17	11.8	102	92	94	0.087	2.68	1621
161	0.65	0.05	0.24	1.58	12.2	103	92	92	0.063	2.5	1626
162	0.69	0.02	0.25	1.11	13.1	109	91	95	0.055	2.59	1628
163	0.68	0.05	0.49	1.3	9.6	94	90	85	0.11	2.78	1637
164	0.64	0.03	0.28	1.44	12.5	97	93	97	0.063	2.51	1638
165	0.66	0.03	0.3	1.57	10.9	103	90	94	0.055	2.62	1642
166	0.63	0.08	0.25	1.26	10.8	98	90	85	0.109	2.73	1642
167	0.65	0.08	0.24	0.82	12.6	102	91	90	0.061	2.51	1651
168	0.65	0.06	0.29	1.23	10.6	96	90	97	0.065	2.65	1655
169	0.68	0.03	0.35	1.31	10.5	98	92	95	0.104	2.76	1656
170	0.62	0.05	0.31	2.06	11	78	90	89	0.079	2.67	1664
171	0.65	0.03	0.31	0.85	14	107	92	98	0.06	2.54	1670
172	0.78	0.07	0.18	1.75	10.4	93	90	98	0.078	2.66	1674
173	0.64	0.04	0.52	1.2	11.9	87	91	80	0.13	2.77	1678
174	0.66	0.11	0.55	1.23	11.4	87	90	88	0.13	2.73	1682
175	0.63	0.01	0.31	1.72	10.1	102	90	99	0.084	2.68	1685
176	0.68	0.08	0.38	0.8	15	97	92	92	0.06	2.5	1686
177	0.67	0.05	0.35	1.8	9.8	96	90	95	0.089	2.65	1697
178	0.64	0.07	0.23	0.91	11.5	92	92	92	0.069	2.5	1704
179	0.8	0.02	0.35	2.31	9.5	89	89	86	0.1	2.64	1705
180	0.8	0.07	0.34	2	9.9	94	89	104	0.08	2.63	1711
181	0.63	0.21	0.54	1.27	9	89	92	86	0.135	2.73	1714
182	0.63	0.03	0.28	2.62	10.5	81	91	91	0.101	2.65	1722
183	0.62	0.04	0.21	0.91	12.6	100	92	94	0.068	2.52	1725
184	0.62	0.15	0.44	1.4	11.6	89	91	94	0.153	2.72	1732
185	0.62	0.04	0.23	1.38	10.8	96	91	83	0.133	2.75	1734
186	0.73	0.04	0.21	1.5	10.9	97	89	94	0.044	2.58	1744
187	0.6	0.03	0.25	0.89	12.7	96	92	94	0.056	2.49	1744
188	0.63	0.03	0.22	1.3	11.3	90	91	89	0.129	2.73	1746
189	0.73	0.03	0.22	1.5	10.2	112	89	90	0.045	2.6	1753
190	0.63	0.01	0.23	1.59	6.6	100	92	90	0.1	2.79	1757
191	0.64	0.03	0.16	1.8	12.4	100	92	83	0.068	2.58	1764
192	0.74	0.03	0.22	1.5	10.7	94	91	99	0.05	2.59	1768
193	0.67	0.02	0.23	1.7	10.9	106	91	96	0.061	2.55	1810
194	0.66	0	0.36	1.3	12.5	82	91	89	0.131	2.82	1817
195	0.62	0.02	0.24	1.3	11.5	81	94	89	0.134	2.75	1818

Table B2. Raw data for ASTM physical testing

	A	B	C	D	E	F	G
1	Sample	M.C.	LOI	Fineness	7Day	Autoclave	S.G.
2	OTT032283	0.07	0.19	9.8		0.075	2.57
3	OTT041383	0.12	0.23	9.2		0.071	2.65
4	OTT042783	0.042	0.21	10.7		0.068	2.65
5	OTT050483	0.028	0.2	8.8		0.08	2.65
6	OTT050983	0.024	0.26	9.4		0.079	2.62
7	OTT051183	0.03	0.32	10.5		0.064	2.59
8	OTT051783	0.05	0.28	10.7		0.067	2.64
9	OTT052783	0.04	0.27	12		0.065	2.66
10	OTT060583	0.03	0.18	11.1		0.068	2.64
11	OTT061083	0.09	0.17	8.8		0.076	2.61
12	OTT061483	0.06	0.13	12.5		0.07	2.66
13	OTT062283	0.038	0.25	10.5		0.055	2.64
14	OTT062883	0.1	0.32	9.9		0.045	2.66
15	OTT063083	0.09	0.32	9		0.057	2.64
16	OTT070783	0.06	0.37	9.6		0.066	2.63
17	OTT071383	0.04	0.16	10.4		0.065	2.6
18	OTT071583	0.05	0.21	9.9		0.049	2.61
19	OTT071983	0.07	0.3	12		0.07	2.59
20	OTT072583	0.05	0.19	9.8		0.05	2.65
21	OTT080183	0.04	0.2	10.7		0.057	2.67
22	OTT080583	0.05	0.54	9.3		0.052	2.64
23	OTT081083	0.04	0.22	12.1		0.052	2.66
24	OTT081583	0.01	0.23	11.1		0.057	2.6
25	OTT081883	0.02	0.28	11.6		0.066	2.63
26	OTT082383	0.01	0.23	11.2		0.052	2.57
27	OTT082683	0.017	0.23	10.4		0.046	2.58
28	OTT090183	0.047	0.15	9.6		0.035	2.64
29	OTT090683	0.11	0.15	9.7		0.048	2.59
30	OTT091283	0.092	0.17	10.2		0.058	2.57
31	OTT091583	0.051	0.21	10		0.065	2.59
32	OTT092283	0.074	0.26	10.2		0.05	2.64
33	OTT092983	0.1	0.21	8.8		0.064	2.59
34	OTT100483	0.122	0.15	10.7		0	2.58
35	OTT110783	0.12	0.1	9.4		0.008	2.59
36	OTT101083	0.112	0.12	10.4		0.003	2.5
37	OTT121283	0.034	0.21	10.5		0.006	2.58
38	OTT111883	0.023	0.24	10.2		0.007	2.55
39	OTT101983	0.12	0.22	10.9		0.007	2.54
40	OTT103183	0.11	0.1	10.6		0.01	2.59
41	OTT012084	0.019	0.221	10		0.005	2.59
42	OTT021384	0.028	0.193	8.3		-0.01	2.62
43	OTT030584	0.02	0.22	10.2	129	0.011	2.56
44	OTT040384	0.03	0.202	12.4	101	0.004	2.5
45	OTT041884	0.019	0.227	12.1	112	0.034	2.57
46	OTT042084	0.017	0.26	11.6	94	0.04	2.52
47	OTT042684	0.008	0.201	10	94	0.04	2.62

Table B2. (Continued)

	A	B	C	D	E	F	G
48	CBF050384	0.05	0.64	12.7	103	0.052	2.65
49	OTT050384	0.01	0.159	9.8	95	0.04	2.6
50	OTT050784	0.01	0.125	9.5	94	0.011	2.61
51	OTT051084	0.039	0.403	9.3	102	0.031	2.63
52	OTT051484	0.021	0.395	9.2	95	0.03	2.57
53	OTT051684	0.045	0.244	8.9	91	0.027	2.62
54	OTT051884	0.006	0.2	10	91	0.034	2.61
55	OTT052184	0.029	0.214	9.5	90	0.034	2.63
56	OTT052384	0.015	0.222	8.8	98	0.026	2.67
57	OTT052584	0.005	0.235	8.9	100	0.025	2.61
58	OTT053184	0.026	0.128	10	93	0.018	2.62
59	OTT060284	0.025	0.261	9.3	100	0.024	2.59
60	OTT060584	0.027	0.263	9.5	94	0.028	2.61
61	OTT060884	0.044	0.273	9	99	0.026	2.6
62	OTT061384	0.049	0.188	10.8	102	0.031	2.62
63	OTT061984	0.015	0.202	9.3	95	0.031	2.67
64	OTT062284	0.015	0.181	9.3	97	0.031	2.68
65	OTT062384	0.013	0.175	9.5	95	0.029	2.67
66	OTT062684	0.016	0.19	9.9	96	0.034	2.65
67	OTT062784	0.006	0.208	9.2	95	0.03	2.65
68	OTT063084	0.019	0.273	10.2	94	0.026	2.63
69	OTT070384	0.023	0.201	9.4	93	0.022	2.66
70	OTT070984	0.028	0.232	9.7	84	0.026	2.64
71	CBF071084	0.059	0.314	10.6	99	0.068	2.69
72	OTT071184	0.022	0.241	9.8	93	0.038	2.62
73	OTT071684	0.015	0.284	9.6	103	0.041	2.62
74	OTT071884	0.013	0.283	11.6	83	0.03	2.6
75	OTT072084	0.01	0.175	10.7	88	0.036	2.58
76	OTT072184	0.014	0.192	10.9	90	0.042	2.58
77	OTT072484	0.013	0.17	10.7	93	0.034	2.57
78	OTT072584	0.019	0.18	10.8	91	0.037	2.58
79	CBF072784	0.02	0.41	11.6	92	0.081	2.72
80	OTT072884	0.02	0.33	11.7	90	0.022	2.59
81	OTT073184	0.033	0.268	11.1	88	0.02	2.58
82	OTT080184	0.025	0.27	11.9	89	0.028	2.58
83	OTT080284	0.028	0.326	11.1	93	0.031	2.56
84	OTT080384	0.025	0.302	11.7	89	0.032	2.55
85	OTT080684	0.032	0.187	11.3	93	0.036	2.57
86	OTT080784	0.027	0.321	13.3	91	0.037	2.57
87	CBF080984	0.01	0.458	12	85	0.116	2.68
88	OTT080984	0.012	0.299	11.9	93	0.031	2.59
89	CBF081084	0.01	0.555	12.2	88	0.092	2.69
90	LAN081084	0.04	0.329	7.7	92	0.086	2.82
91	OTT081084	0.002	0.255	11.3	88	0.028	2.63
92	OTT081384	0.023	0.253	10.7	99	0.028	2.62
93	OTT081484	0.016	0.266	12	92	0.029	2.57
94	CBF081584	0.03	0.704	13	92	0.057	2.73

Table B2. (Continued)

	A	B	C	D	E	F	G
95	OTT081584	0.008	0.274	11.3	97	0.032	2.55
96	OTT081684	0.008	0.301	11	93	0.032	2.56
97	LAN081784	0.033	0.324	7.1	95	0.105	2.82
98	OTT081784	0	0.333	9.6	89	0.032	2.54
99	CBF081884	0.013	0.296	12	99	0.049	2.69
100	OTT082084	0.009	0.318	10.2	100	0.037	2.56
101	OTT082284	0.019	0.305	9.4	91	0.035	2.6
102	OTT082384	0.002	0.199	10.1	99	0.033	2.6
103	CBF082484	0.04	0.362	10.9	90	0.062	2.67
104	OTT082584	0.002	0.204	9.2	83	0.038	2.62
105	OTT082784	0.011	0.158	10.5	85	0.036	2.6
106	OTT082884	0.004	0.197	10.2	83	0.036	2.61
107	OTT082984	0.004	0.233	10.2	84	0.038	2.6
108	OTT083084	0.021	0.202	10.3	74	0.036	2.61
109	CBF090184	0.076	0.426	10.8	94	0.078	2.7
110	LAN090484	0.147	0.421	9.4	98	0.074	2.8
111	OTT090484	0.024	0.192	10	82	0.042	2.61
112	CBF090584	0.073	0.424	12.4	85	0.067	2.69
113	OTT090584	0.008	0.179	10.7	84	0.044	2.59
114	CBF090684	0.043	0.454	13.8	98	0.06	2.71
115	CBF090784	0.234	0.538	14.3	95	0.06	2.72
116	OTT090784	0.01	0.244	10.5	94	0.034	2.62
117	NE4091084	0.01	0.259	11.8	92	0.064	2.64
118	CBF091184	0.029	0.305	9.9	94	0.052	2.54
119	LAN091284	0.062	0.347	8.3	84	0.082	2.78
120	NE4091284	0.039	0.279	11.3	89	0.07	2.66
121	OTT091284	0.042	0.384	9.1	86	0.03	2.61
122	CBF091484	0.117	0.528	14.4	87	0.052	2.57
123	CBF091684	0.022	0.405	11.1	89	0.076	2.64
124	CBF091784	0.173	0.643	11.6	87	0.056	2.56
125	CBF091884	0.024	0.435	10.6	98	0.054	2.63
126	NE4091984	0.01	0.363	13.3	86	0.074	2.66
127	NE4092084	0.034	0.42	12	87	0.08	2.65
128	CBF092184	0.023	0.264	11.1	94	0.078	2.6
129	NE4092284	0.004	0.376	11.2	87	0.08	2.63
130	CBF092484	0.012	0.248	11.6	87	0.08	2.57
131	NE4092484	0.003	0.224	8.4	86	0.078	2.65
132	CBF092684	0.104	0.509	13.9	79	0.082	2.62
133	OTT092684	0.048	0.23	10.8	79	0.043	2.58
134	NE4092784	0.006	0.286	13.3	95	0.065	2.67
135	CBF092884	0.012	0.38	13	91	0.086	2.59
136	LAN092984	0.021	0.196	10.2	80	0.056	2.76
137	NE4092984	0.004	0.272	12	89	0.074	2.66
138	CBF100284	0.056	0.592	13.3	89	0.086	2.66
139	NE4100284	0.035	0.303	9.4	81	0.074	2.67
140	OTT100284	0.005	0.229	11.2	95	0.038	2.48
141	OTT100384	0.003	0.244	12.5	82	0.028	2.47

Table B2. (Continued)

	A	B	C	D	E	F	G
142	CBF100484	0.021	0.522	12.2	74	0.094	2.64
143	LAN100484	0.013	0.21	10.4	92	0.048	2.73
144	OTT100584	0.004	0.237	11.9	96	0.028	2.48
145	NE4100684	0.014	0.317	11.4	82	0.053	2.62
146	OTT100884	0.017	0.434	10.6	83	0.032	2.55
147	OTT101084	0.005	0.382	12.2	82	0.043	2.56
148	CBF101184	0.022	0.574	15.8	86	0.079	2.67
149	LAN101284	0.075	0.312	9.7	91	0.05	2.76
150	OTT101284	0.018	0.271	13	82	0.034	2.57
151	OTT101784	0.032	0.232	10.7	87	0.044	2.59
152	LAN101884	0.08	0.206	10.3	85	0.066	2.76
153	CBF102284	0.029	0.542	13.7	101	0.092	2.67
154	LAN102684	0.053	0.134	10.3	86	0.062	2.78
155	CBF102984	0.027	0.476	13.5	99	0.077	2.64
156	OTT102984	0.039	0.187	10.2	84	0.044	2.6
157	LAN110284	0.083	0.248	11	88	0.066	2.8
158	OTT110584	0.024	0.252	11.3	92	0.027	2.52
159	NE4110784	0.064	0.281	9.9	97	0.066	2.56
160	OTT110884	0.022	0.227	10.6	84	0.034	2.57
161	LAN110984	0.072	0.238	8.6	79	0.058	2.75
162	OTT111484	0.026	0.272	11.1	100	0.042	2.61
163	NE4111584	0.081	0.292	9	79	0.056	2.64
164	OTT112084	0.03	0.209	10	96	0.052	2.61
165	NE4112484	0.02	0.336	12.8	90	0.049	2.66
166	CBF112684	0.04	0.527	14.4	96	0.083	2.71
167	LAN112684	0.055	0.286	10.1	81	0.088	2.8
168	OTT112884	0.024	0.364	10.3	81	0.042	2.62
169	LAN120184	0.025	0.266	8.9	87	0.077	2.8
170	NE4121084	0.036	0.232	11.1	87	0.041	2.62
171	OTT121084	0.027	0.23	9.8	95	0.03	2.63
172	OTT122084	0.02	0.203	10.3	92	0.021	2.62
173	OTT011385	0.013	0.15	11.6	89	0.04	2.58
174	OTT022085	0.019	0.224	12.3	105	0.058	2.61
175	OTT031585	0.011	0.176	11.4	109	0.074	2.6
176	OTT032685	0.015	0.299	11.2	88	0.068	2.61
177	OTT040385	0.007	0.28	11.1	77	0.078	2.67
178	OTT041085	0.012	0.126	11.1	84	0.087	2.65
179	OTT041285	0.002	0.111	10	90	0.076	2.64
180	OTT041685	0.003	0.178	10.9	91	0.082	2.64
181	OTT041885	0.004	0.14	10.5	98	0.09	2.58
182	OTT041985	0	0.18	9.8	90	0.084	2.62
183	NE4042285	0.02	0.167	15.4	95	0.099	2.63
184	OTT042485	0.016	0.172	10.4	87	0.083	2.64
185	NE4043085	0.04	0.326	13.9	88	0.1	2.68
186	NE4050185	0.02	0.283	14.4	90	0.092	2.66
187	OTT050185	0.05	0.262	12.1	92	0.054	2.68
188	OTT050285	0.01	0.257	10.4	86	0.058	2.7

Table B2. (Continued)

	A	B	C	D	E	F	G
189	OTT050385	0.04	0.228	9.5	93	0.045	2.63
190	OTT050785	0.02	0.196	10	93	0.052	2.62
191	OTT050885	0.01	0.239	9.9	92	0.066	2.68
192	OTT051085	0.01	0.183	9.8	96	0.067	2.67
193	NE4051185	0.01	0.3	16.3	88	0.06	2.7
194	LAN051385	0.04	0.318	15.1	87	0.117	2.81
195	OTT051485	0.02	0.226	10.7	86	0.05	2.74
196	OTT051685	0.01	0.206	9	89	0.05	2.69
197	LAN052085	0.03	0.268	13.3	85	0.124	2.79
198	NE4052085	0.02	0.231	15.3	91	0.076	2.69
199	OTT052085	0.01	0.234	9.6	91	0.061	2.67
200	OTT052185	0.01	0.18	9.8	86	0.069	2.68
201	NE4052285	0.01	0.223	15.4	99	0.08	2.69
202	OTT052385	0.01	0.247	8.7	104	0.069	2.7
203	CBF052485	0.07	0.463	15.6	99	0.09	2.69
204	CBF052485	0.07	0.463	15.6	99	0.09	2.69
205	OTT052885	0.03	0.222	9.6	98	0.068	2.68
206	NE4052985	0.02	0.22	14.1	94	0.077	2.64
207	OTT053085	0.03	0.157	9.2	101	0.072	2.7
208	OTT060185	0.01	0.187	9.5	95	0.072	2.7
209	NE4060385	0.02	0.208	13.5	91	0.1	2.61
210	OTT060485	0.01	0.177	10.7	93	0.075	2.7
211	OTT060585	0.02	0.125	9.8	102	0.071	2.68
212	OTT060685	0.02	0.219	10.4	96	0.074	2.67
213	CBF060785	0.6	1.58	18.3	92	0.096	2.67
214	NE4060785	0.03	0.161	13.6	89	0.089	2.61
215	OTT060785	0.01	0.186	9.8	96	0.073	2.69
216	OTT060885	0.02	0.268	9.8	90	0.071	2.68
217	OTT061085	0.03	0.17	11.4	86	0.073	2.62
218	NE4061285	0.04	0.279	15.3	93	0.092	2.64
219	OTT061285	0.05	0.15	11	94	0.064	2.64
220	CBF061385	0.28	0.956	13.4	88	0.11	2.66
221	OTT061385	0.02	0.141	11	93	0.064	2.63
222	LAN061585	0.02	0.768	12.4	91	0.121	2.82
223	OTT061785	0.08	0.169	10.2	100	0.076	2.66
224	OTT061885	0.03	0.146	10.1	91	0.066	2.65
225	NE4061985	0.04	0.467	10.6	92	0.092	2.6
226	OTT061985	0.03	0.284	8.8	89	0.069	2.65
227	OTT062185	0.1	0.284	9.7	92	0.07	2.62
228	CBF062585	0.12	0.497	12.8	87	0.117	2.71
229	LAN062585	0.04	0.63	11.9	85	0.114	2.81
230	NE4062585	0.08	0.472	10.2	84	0.093	2.6
231	OTT062585	0.02	0.299	9.3	98	0.074	2.6
232	OTT062685	0.02	0.304	9.4	96	0.073	2.63
233	OTT062885	0.03	0.246	9.7	82	0.075	2.65
234	CBF070185	0.01	0.303	12.7	77	0.122	2.75
235	OTT070185	0.03	0.234	9.7	85	0.076	2.58

Table B2. (Continued)

	A	B	C	D	E	F	G
236	NE4070285	0.05	0.364	10.1	88	0.1	2.6
237	OTT070385	0.02	0.206	10	90	0.07	2.58
238	LAN070685	0.06	0.519	12.7	84	0.129	2.79
239	NE4070685	0.02	0.302	10.6	80	0.097	2.61
240	CBF070885	0.09	0.599	13.8	80	0.132	2.74
241	NE4070885	0.02	0.269	10.5	90	0.083	2.53
242	OTT070885	0.03	0.288	9.4	90	0.074	2.58
243	NE4071285	0.04	0.337	11.5	83	0.093	2.58
244	OTT071285	0.03	0.263	10.1	97	0.09	2.65
245	LAN071385	0.03	0.454	12.3	86	0.117	2.8
246	CBF071585	0.05	0.358	12.8	84	0.142	2.76
247	NE4071885	0.03	0.312	13.1	90	0.091	2.57
248	OTT071985	0.04	0.335	10.2	94	0.075	2.68
249	LAN072085	0.05	0.481	12.2	85	0.136	2.8
250	NE4072085	0.04	0.349	11.8	86	0.092	2.52
251	CBF072385	0.08	0.494	14.3	91	0.127	2.73
252	NE4072385	0.02	0.327	10.9	89	0.09	2.62
253	OTT072385	0.05	0.285	9.5	92	0.062	2.69
254	OTT072685	0.02	0.286	8.8	98	0.075	2.65
255	LAN072785	0.07	0.414	10.2	85	0.119	2.79
256	NE4072785	0.02	0.329	12.7	86	0.095	2.57
257	CBF072985	0.3	0.704	12.8	86	0.062	2.68
258	OTT073085	0.04	0.266	9.8	94	0.068	2.69
259	NE4073185	0.04	0.379	11.7	93	0.08	2.64
260	OTT080185	0.02	0.273	9.4	93	0.066	2.68
261	LAN080285	0.01	0.3	11.1	84	0.11	2.81
262	OTT080285	0.05	0.197	8.4	91	0.042	2.68
263	NE4080585	0.05	0.318	11.3	86	0.06	2.61
264	OTT080585	0.01	0.185	9.1	87	0.052	2.65
265	CBF080685	0.2	0.544	13.8	92	0.078	2.72
266	OTT080685	0.03	0.351	9.4	94	0.05	2.64
267	NE4080785	0.03	0.255	10.4	102	0.067	2.64
268	OTT080785	0.04	0.183	10.2	94	0.047	2.63
269	OTT080885	0.02	0.211	9.8	96	0.046	2.62
270	NE4080985	0.03	0.272	10.3	97	0.07	2.61
271	OTT080985	0.03	0.218	9.6	96	0.034	2.62
272	LAN081085	0.04	0.537	14.2	92	0.094	2.8
273	NE4081285	0.06	0.303	10.4	86	0.07	2.6
274	OTT081285	0.03	0.26	8.6	109	0.032	2.64
275	OTT081485	0.04	0.249	9.6	95	0.044	2.67
276	NE4081585	0.17	0.405	11.4	89	0.074	2.68
277	OTT081585	0.02	0.255	9.3	96	0.054	2.67
278	CBF081685	0.05	0.435	11	94	0.104	2.72
279	NE4081785	0.02	0.353	10	86	0.054	2.65
280	NE4081985	0.08	0.391	8.8	86	0.069	2.64
281	OTT081985	0.03	0.264	8.8	97	0.054	2.67
282	OTT082085	0.02	0.259	10.5	91	0.042	2.66

Table B2. (Continued)

	A	B	C	D	E	F	G
283	NE4082185	0.05	0.282	9.8	93	0.068	2.68
284	NE4082385	0.04	0.272	8	91	0.075	2.67
285	LAN082485	0.05	0.585	11.7	85	0.112	2.81
286	NE4082485	0.08	0.452	8.7	96	0.07	2.66
287	NE4082685	0.07	0.313	8.5	98	0.068	2.65
288	OTT082685	0.05	0.263	9.9	97	0.058	2.65
289	CBF082785	0.09	0.509	10.6	93	0.095	2.7
290	NE4082785	0.03	0.32	8.7	99	0.069	2.67
291	OTT082785	0.06	0.278	8.7	103	0.054	2.64
292	NE4082885	0.03	0.241	8.5	97	0.064	2.66
293	CBF083085	0.05	0.254	12.3	84	0.095	2.7
294	NE4083085	0.04	0.328	9.6	96	0.072	2.61
295	OTT083085	0.04	0.31	9.9	94	0.044	2.62
296	NE4090485	0.02	0.219	13.5	89	0.066	2.63
297	OTT090585	0.02	0.351	8.5	99	0.044	2.63
298	CBF090685	0.03	0.268	13.4	87	0.104	2.7
299	LAN090785	0.02	0.335	9.7	84	0.089	2.78
300	NE4090885	0.02	0.311	12.3	91	0.076	2.65
301	CBF090985	0.3	0.569	13.9	87	0.103	2.7
302	NE4091085	0.04	0.33	12.3	90	0.077	2.61
303	OTT091085	0.02	0.277	9.1	94	0.042	2.66
304	CBF091185	0.32	0.474	14.6	87	0.112	2.71
305	NE4091185	0.04	0.303	11.8	87	0.074	2.58
306	NE4091285	0.09	0.292	8.4	86	0.078	2.55
307	OTT091285	0.01	0.287	9.3	88	0.03	2.62
308	NE4091385	0.07	0.378	9.1	77	0.071	2.56
309	CBF091585	0.08	0.273	9.8	84	0.108	2.74
310	CBF091685	0.07	0.235	9.3	90	0.114	2.74
311	NE4091785	0.07	0.359	9.2	100	0.047	2.59
312	CBF091885	0.11	0.342	12.1	89	0.083	2.74
313	NE4091885	0.07	0.32	11.9	102	0.05	2.61
314	NE4091985	0.05	0.317	8.5	98	0.06	2.54
315	CBF092085	0.08	0.334	10.7	86	0.089	2.72
316	OTT092085	0.03	0.269	10.4	88	0.052	2.64
317	LAN092185	0.09	0.529	12.3	94	0.096	2.75
318	NE4092185	0.16	0.318	9	82	0.066	2.53
319	OTT092385	0.02	0.27	9.1	98	0.037	2.67
320	OTT092585	0.01	0.266	9.2	92	0.061	2.68
321	LAN092785	0.15	0.578	9.3	95	0.088	2.76
322	NE4092785	0.03	0.307	9	99	0.066	2.54
323	CBF093085	0.04	0.246	10.2	89	0.096	2.7
324	OTT093085	0.01	0.259	9.3	90	0.04	2.67
325	NE4100185	0.02	0.307	9.7	100	0.046	2.49
326	OTT100385	0.01	0.229	10.2	96	0.049	2.65
327	NE4100485	0.02	0.265	11.9	101	0.033	2.37
328	OTT100485	0.03	0.291	10.4	95	0.046	2.62
329	NE4100885	0.01	0.192	12.9	102	0.026	2.36

Table B2. (Continued)

	A	B	C	D	E	F	G
330	OTT100885	0.01	0.247	10.4	89	0.043	2.64
331	NE4101085	0.02	0.266	10.5	98	0.016	2.42
332	OTT101085	0.04	0.282	8.8	95	0.047	2.63
333	CBF101185	0.05	0.324	7.8	89	0.081	2.65
334	LAN101185	0.06	0.417	14.3	80	0.049	2.72
335	OTT101285	0.02	0.327	9	100	0.04	2.66
336	NE4101485	0.05	0.305	11.5	101	0.025	2.47
337	OTT101585	0.02	0.309	8.7	100	0.045	2.65
338	NE4101685	0.03	0.277	10.5	99	0.031	2.45
339	OTT101785	0.04	0.284	9.6	100	0.047	2.62
340	NE4101985	0.03	0.351	12	98	0.024	2.43
341	NE4101985	0.03	0.351	12	98	0.024	2.43
342	CBF102185	0.09	0.346	9.7	93	0.065	2.62
343	OTT102285	0.05	0.26	9.6	97	0.042	2.65
344	OTT102585	0.03	0.236	9.4	93	0.047	2.67
345	OTT102885	0.03	0.216	9	95	0.058	2.66
346	OTT103185	0.03	0.205	9.6	99	0.051	2.64
347	OTT110485	0.02	0.301	9.5	91	0.038	2.63
348	OTT110685	0.05	0.29	10	96	0.047	2.63
349	OTT111185	0.05	0.276	9.3	93	0.046	2.61
350	OTT112285	0.05	0.266	9.3	100	0.021	2.63
351	OTT040386	0.06	0.255	8.6	102	0.056	2.68
352	LAN040786	0	0.957	14.9	96	0.115	2.76
353	OTT041286	0.05	0.42	8.5	109	0.07	2.68
354	CBF041886	0.08	0.292	10.5	102	0.09	2.66
355	OTT042186	0.03	0.257	8.7	105	0.061	2.68
356	LAN042586	0.05	0.43	13.9	95	0.114	2.78
357	OTT042886	0.1	0.307	8.8	107	0.064	2.69
358	CBF043086	0.07	0.242	10.5	95	0.109	2.68
359	OTT050186	0.03	0.268	10.3	107	0.08	2.68
360	CBF050686	0.05	0.277	7.6	97	0.121	2.73
361	OTT050786	0.04	0.316	8.4	106	0.073	2.75
362	CBF050986	0.03	0.239	7.6	89	0.134	2.73
363	OTT050986	0.03	0.266	9	111	0.05	2.68
364	OTT051386	0.05	0.236	8.7	106	0.057	2.69
365	NE4051586	0.06	0.372	9.8	98	0.106	2.7
366	OTT051686	0.02	0.326	9.1	102	0.084	2.67
367	CBF051986	0.1	0.571	9.8	84	0.145	2.71
368	LAN052086	0.04	0.754	13.7	92	0.174	2.8
369	NE4052086	0.02	0.343	11.4	91	0.107	2.69
370	OTT052186	0.02	0.293	7.4	99	0.068	2.72
371	NE4052386	0.03	0.313	12.4	95	0.11	2.69
372	OTT052386	0.02	0.333	8.1	104	0.05	2.72
373	CBF052786	0.02	0.294	9.4	99	0.11	2.71
374	NE4052886	0.02	0.264	11.8	79	0.111	2.7
375	OTT052886	0.01	0.376	9.3	103	0.067	2.7
376	CBF053086	0.02	0.315	8.8	88	0.13	2.7

Table B2. (Continued)

	A	B	C	D	E	F	G
377	NE4053186	0.02	0.276	10.4	82	0.094	2.7
378	OTT053186	0.01	0.235	8.6	96	0.063	2.7
379	NE4060386	0.02	0.39	11.9	86	0.125	2.7
380	OTT060486	0.02	0.355	9.1	100	0.07	2.7
381	CBF060586	0.02	0.208	8.5	98	0.12	2.72
382	NE4060686	0.05	0.403	11.1	86	0.129	2.72
383	OTT060686	0.02	0.394	8.3	91	0.069	2.68
384	NE4060886	0.05	0.389	10.7	86	0.108	2.7
385	CBF060986	0.16	0.66	10.2	92	0.104	2.68
386	NE4061086	0.04	0.414	12	86	0.104	2.71
387	NE4061286	0.03	0.391	12.8	97	0.105	2.72
388	OTT061286	0.04	0.366	8.6	90	0.066	2.71
389	OTT061386	0.02	0.293	8.6	93	0.068	2.71
390	NE4061486	0.05	0.399	12.8	77	0.104	2.73
391	CBF061686	0.05	0.258	9	90	0.127	2.7
392	NE4061686	0.09	0.397	12.2	95	0.107	2.7
393	NE4061786	0.08	0.473	12.5	92	0.106	2.71
394	OTT061786	0.08	0.279	8.2	98	0.082	2.7
395	NE4061986	0.03	0.324	11.2	100	0.117	2.73
396	OTT061986	0.09	0.324	8.4	95	0.087	2.69
397	NE4062186	0.04	0.264	12	95	0.106	2.73
398	CBF062386	0.12	0.437	10.6	84	0.112	2.7
399	OTT062386	0.05	0.367	9.1	88	0.093	2.7
400	NE4062486	0.03	0.384	11.6	89	0.127	2.7
401	OTT062486	0.05	0.286	8.9	93	0.088	2.71
402	CBF062686	0.07	0.324	10.3	93	0.104	2.72
403	OTT062686	0.05	0.283	9.2	91	0.096	2.69
404	NE4062786	0.02	0.339	12.4	85	0.115	2.68
405	NE4062986	0.04	0.39	11.5	88	0.129	2.68
406	CBF070186	0.04	0.427	10.7	92	0.09	2.72
407	OTT070186	0.02	0.304	9.5	97	0.087	2.69
408	LAN070286	0.02	0.573	10.7	92	0.13	2.8
409	NE4070286	0.08	0.44	12	89	0.116	2.69
410	OTT070386	0.03	0.271	8.8	95	0.091	2.7
411	NE4070786	0.04	0.36	12.5	85	0.108	2.68
412	OTT070786	0.05	0.364	9.3	86	0.099	2.7
413	NE4070886	0.08	0.284	11.4	86	0.101	2.72
414	CBF071086	0.08	0.384	10.7	91	0.032	2.72
415	OTT071086	0.13	0.215	9.1	91	0.073	2.71
416	NE4071186	0.02	0.295	11.7	89	0.029	2.7
417	NE4071386	0.17	0.4	12.8	78	0.033	2.71
418	LAN071486	0.09	0.56	13.9	74	0.121	2.8
419	NE4071586	0.03	0.333	11.5	92	0.035	2.7
420	NE4071686	0.02	0.249	13.2	90	0.031	2.69
421	OTT071686	0.04	0.308	9.4	90	0.002	2.71
422	NE4071786	0.07	0.315	13.1	89	0.026	2.67
423	CBF071886	0.03	0.508	10.8	84	0.093	2.68

Table B2. (Continued)

	A	B	C	D	E	F	G
424	NE4071986	0.07	0.349	13.2	84	0.029	2.67
425	LAN072186	0.07	0.53	11.9	82	0.062	2.8
426	NE4072186	0.08	0.337	11.4	88	0.023	2.68
427	OTT072186	0.08	0.299	9.2	88	0.014	2.7
428	NE4072386	0.02	0.269	12.2	77	0.018	2.67
429	CBF072486	0.03	0.399	11.8	86	0.075	2.74
430	NE4072486	0.03	0.239	11.7	83	0.017	2.7
431	OTT072486	0.04	0.399	9.2	84	0.011	2.69
432	NE4072586	0.03	0.264	10.2	80	0.024	•
433	OTT072586	0.02	0.409	9.4	81	0.012	2.71
434	LAN073086	0.04	0.737	12.1	87	0.064	2.8
435	OTT073086	0.11	0.444	9.6	89	0.005	2.7
436	OTT073186	0.05	0.35	9.6	88	0.008	2.71
437	CBF080186	0.07	0.534	11.7	77	0.104	2.71
438	OTT080486	0.02	0.344	10.3	96	0.005	2.68
439	OTT080786	0.03	0.17	9.4	93	0.003	2.63
440	CBF080886	0.07	0.344	10.6	93	0.11	2.74
441	LAN081186	0.07	0.434	10.2	85	0.047	2.79
442	OTT081186	0.03	0.169	10.4	86	0.007	2.64
443	CBF081286	0.2	0.448	9.5	80	0.106	2.73
444	OTT081386	0.01	0.227	10.6	86	0.008	2.65
445	OTT081486	0.03	0.194	8.7	96	0.007	2.66
446	LAN081886	0.02	0.428	10	86	0.044	2.8
447	OTT081886	0.03	0.31	9.8	90	0.006	2.66
448	CBF081986	0.08	0.521	9.7	83	0.088	2.71
449	OTT081986	0.02	0.326	9.4	94	0.01	2.67
450	OTT082086	0.03	0.314	9.2	95	0.001	2.66
451	CBF082286	0.204	0.737	9.1	79	0.07	2.71
452	OTT082286	0.03	0.225	7.9	95	0.002	2.64
453	OTT082386	0.03	0.206	7.9	94	0.009	2.69
454	LAN082586	0.02	0.286	10.2	84	0.049	2.77
455	CBF082686	0.2	0.799	11.2	86	0.058	2.7
456	OTT082786	0.01	0.344	8.8	86	0.007	2.68
457	OTT082886	0.02	0.309	9.5	87	0.005	2.67
458	CBF082986	0.17	0.916	10.2	88	0.059	2.69
459	OTT082986	0	0.326	8.6	93	0.004	2.68
460	OTT090286	0	0.348	9.4	88	0.004	2.69
461	OTT090386	0.01	0.299	9.1	93	0.004	2.7
462	CBF090486	0.03	0.325	7.8	85	0.053	2.7
463	OTT090486	0.03	0.328	10.2	86	0	2.67
464	CBF090586	0.01	0.319	10.9	86	0.05	2.7
465	CBF090686	0.02	0.362	9.6	87	0.048	2.71
466	OTT090686	0	0.346	9.7	94	-0.01	2.69
467	CBF090886	0.03	0.294	10.8	86	0.048	2.69
468	LAN090886	0	0.392	9.5	81	0.055	2.8
469	OTT090886	0.01	0.33	8.9	91	0	2.7
470	OTT090986	0	0.324	9.1	100	0.007	2.69

Table B2. (Continued)

	A	B	C	D	E	F	G
471	CBF091086	0.04	0.41	10.6	88	0.072	2.72
472	OTT091186	0.04	0.343	9.7	91	0.008	2.69
473	OTT091286	0.02	0.351	8.7	97	0.008	2.69
474	LAN091586	0.01	0.305	7.2	91	0.055	2.77
475	OTT091586	0	0.341	10.3	97	0.011	2.69
476	CBF091686	0.11	0.475	9.6	88	0.051	2.63
477	OTT091686	0.01	0.317	10.1	101	0.014	2.69
478	OTT091786	0.01	0.372	10.4	94	0.012	2.68
479	OTT091986	0.01	0.368	10.4	93	0.012	2.68
480	OTT092286	0.03	0.397	11.5	91	0.012	2.69
481	LAN092386	0	0.799	12.9	84	0.051	2.77
482	OTT092486	0.02	0.264	11.2	95	0.011	2.69
483	OTT092686	0.04	0.339	10.4	92	0.007	2.67
484	LAN092986	0.02	0.515	11.7	82	0.065	2.76
485	OTT092986	0.06	0.313	10.3	80	0.011	2.66
486	OTT100286	0.09	0.448	12.1	91	0.004	2.67
487	LAN100686	0	0.203	8.1	85	0.051	2.81
488	OTT100686	0.04	0.386	11.1	99	0.011	2.68
489	OTT100886	0	0.333	11	88	0.015	2.67
490	OTT100986	0.01	0.334	10.9	92	0.012	2.69
491	OTT101086	0	0.329	10	89	0.004	2.66
492	LAN101586	0.01	0.415	9.3	90	0.06	2.8
493	OTT101586	0	0.274	9.6	86	0	2.65
494	OTT101786	0.01	0.293	9.6	89	0.003	2.66
495	OTT102186	0.02	0.261	10.3	90	0.014	2.65
496	LAN102286	0.02	0.431	14.6	80	0.054	2.79
497	OTT102386	0.03	0.255	10	89	0.014	2.65
498	LAN102786	0	0.582	11.8	82	0.067	2.78
499	OTT102986	0.03	0.345	10.3	90	0.013	2.67
500	OTT103186	0	0.239	10.7	85	0.011	2.67
501	LAN110386	0	0.591	13	80	0.069	2.8
502	CBF110586	0.04	0.371	9.8	75	0.059	2.71
503	CBF110686	0.11	0.409	9.8	80	0.062	2.7
504	OTT111086	0.01	0.239	11.1	91	0.011	2.68
505	CBF120886	0.02	0.3	11	85	0.069	2.72
506	OTT121586	0.02	0.243	9.3	83	0.015	2.68
507	OTT.011587	0.05	0.306	9.9	82	0.018	2.67
508	OTT.021587	0	0.292	10.9	83	0.065	2.62
509	OTT.031687	0.03	0.324	10.5	80	0.08	2.67
510	OTT.040287	0.02	0.259	10.6	80	0.049	2.65
511	OTT.040887	0.01	0.338	11.8	95	0.089	2.67
512	OTT.041187	0.02	0.319	11.4	89	0.092	2.63
513	OTT.041587	0.04	0.314	12.1	85	0.105	2.66
514	OTT.041787	0.04	0.335	12	86	0.095	2.68
515	LAN.042087	0.04	0.71	11.2	85	0.136	2.74
516	OTT.042187	0.02	0.37	14.5	82	0.091	2.68
517	OTT.042387	0.01	0.309	10.2	87	0.107	2.64

Table B2. (Continued)

	A	B	C	D	E	F	G
518	OTT.042487	0.02	0.329	9	78	0.105	2.63
519	LAN.042787	0	0.81	15.8	83	0.138	2.72
520	OTT.042887	0.04	0.334	9.7	95	0.104	2.64
521	OTT.042987	0.03	0.333	9.8	89	0.099	2.6
522	LAN.050487	0	0.593	12.9	91	0.142	2.8
523	OTT.050587	0.02	0.354	9.3	95	0.086	2.62
524	OTT.050887	0.01	0.248	9.8	91	0.081	2.64
525	LAN.051087	0.04	0.455	11.6	80	0.147	2.77
526	NE4.051287	0	0.32	17.5	87	0.075	2.54
527	NE4.051287	0.04	0.295	15	91	0.076	2.54
528	NE4.051587	0.03	0.38	13.6	91	0.065	2.52
529	NE4.051887	0.01	0.345	12.6	87	0.077	2.5
530	NE4.051987	0	0.36	13.8	89	0.086	2.54
531	LAN.052087	0	0.37	8.8	93	0.148	2.78
532	NE4.052487	0.02	0.316	12.2	99	0.074	2.53
533	LAN.052687	0.02	0.293	8.8	87	0.125	2.79
534	NE4.052787	0.03	0.324	13.4	92	0.071	2.56
535	NE4.053087	0.03	0.279	12.9	95	0.055	2.52
536	LAN.053187	0.02	0.299	9.9	89	0.113	2.79
537	NE4.060287	0.03	0.274	11.8	85	0.06	2.54
538	NE4.060487	0.02	0.249	11.9	97	0.063	2.54
539	NE4.060687	0.06	0.233	14.7	93	0.059	2.55
540	NE4.060987	0.01	0.259	9.7	94	0.065	2.5
541	OTT.060987	0.02	0.205	12	86	0.038	2.58
542	OTT.061187	0	0.169	12.6	99	0.034	2.54
543	CBF.061287	0.04	0.234	9.2	87	0.095	2.7
544	NE4.061287	0.02	0.244	12.7	88	0.061	2.51
545	NE4.061587	0.01	0.267	13.3	101	0.059	2.52
546	OTT.061587	0.02	0.234	14.3	83	0.037	2.59
547	OTT.061787	0.03	0.324	12.3	88	0.064	2.62
548	NE4.061887	0.06	0.204	12.6	93	0.068	2.52
549	NE4.062187	0.02	0.258	11.3	92	0.066	2.49
550	OTT.062287	0.02	0.351	12.5	96	0.069	2.64
551	NE4.062487	0.04	0.221	12.4	93	0.057	2.5
552	OTT.062487	0.01	0.402	11.4	98	0.073	2.65
553	CBF.062687	0.08	0.205	11.1	92	0.122	2.7
554	NE4.062687	0.06	0.224	13.2	100	0.048	2.51
555	OTT.062787	0.06	0.26	9.9	92	0.063	2.61
556	CBF.062987	0.04	0.207	11	92	0.131	2.74
557	LAN.062987	0.04	0.126	8.5	92	0.098	2.73
558	NE4.062987	0.09	0.284	12.4	96	0.062	2.49
559	OTT.063087	0.08	0.329	11.1	96	0.064	2.61
560	NE4.070187	0.04	0.276	11.5	89	0.071	2.52
561	LAN.070287	0.03	0.289	8.3	89	0.101	2.77
562	NE4.070287	0.01	0.249	11.9	90	0.064	2.54
563	OTT.070287	0.04	0.349	11.1	100	0.061	2.6
564	NE4.070487	0.04	0.282	11.6	90	0.056	2.52

Table B2. (Continued)

	A	B	C	D	E	F	G
565	OTT.070687	0.02	0.276	10.5	88	0.062	2.59
566	CBF.070787	0.1	0.282	10.7	89	0.111	2.74
567	NE4.070787	0.1	0.339	14.5	90	0.048	2.52
568	OTT.070887	0.05	0.304	10.6	97	0.067	2.66
569	NE4.071087	0.07	0.242	12.2	97	0.061	2.49
570	LAN.071387	0	0.401	9.8	89	0.086	2.78
571	OTT.071387	0.07	0.29	10	100	0.057	2.65
572	OTT.071487	0	0.243	10.1	96	0.056	2.67
573	NE4.071687	0.09	0.267	13.8	92	0.061	2.55
574	OTT.071687	0.01	0.226	10.8	93	0.067	2.67
575	CBF.071787	0.05	0.287	11.4	84	0.122	2.73
576	OTT.071787	0.03	0.244	10.4	97	0.062	2.65
577	LAN.072087	0	0.409	10	98	0.082	2.75
578	OTT.072087	0	0.25	12.7	92	0.086	2.68
579	NE4.072187	0.03	0.363	13.9	89	0.068	2.54
580	OTT.072187	0.02	0.254	11.8	88	0.092	2.7
581	CBF.072287	0.14	0.324	11	82	0.142	2.76
582	OTT.072287	0	0.256	11.2	84	0.095	2.66
583	OTT.072387	0.03	0.316	11.5	87	0.086	2.68
584	CBF.072487	0.02	0.371	10.7	88	0.123	2.73
585	NE4.072487	0.02	0.413	15.3	96	0.061	2.53
586	OTT.072487	0.03	0.333	10.2	92	0.092	2.66
587	CBF.072787	0.16	0.285	11	90	0.125	2.73
588	LAN.072887	0	0.541	13.4	98	0.105	2.77
589	NE4.072887	0.02	0.273	14.3	94	0.069	2.53
590	OTT.072887	0.09	0.248	11.2	93	0.088	2.69
591	OTT.073087	0.08	0.251	10.7	103	0.068	2.66
592	NE4.073187	0.04	0.249	15.2	93	0.071	2.54
593	LAN.080387	0.02	0.551	12.5	91	0.111	2.79
594	OTT.080387	0.03	0.226	10.9	99	0.082	2.69
595	NE4.080487	0.06	0.279	11.5	92	0.062	2.5
596	OTT.080487	0.02	0.239	10.2	91	0.085	2.68
597	OTT.080687	0.02	0.203	10	97	0.081	2.66
598	LAN.080787	0.1	0.378	12.6	91	0.105	2.79
599	NE4.080787	0.01	0.24	14.2	95	0.058	2.52
600	OTT.080787	0	0.277	9.8	99	0.079	2.68
601	NE4.081187	0.05	0.281	14.6	94	0.06	2.49
602	OTT.081187	0.03	0.272	10.1	97	0.078	2.71
603	OTT.081287	0.01	0.234	10.4	95	0.065	2.7
604	NE4.081387	0.06	0.242	15.3	90	0.051	2.52
605	OTT.081387	0.02	0.257	10.4	95	0.062	2.65
606	NE4.081787	0.02	0.341	15.6	88	0.055	2.53
607	OTT.081787	0.01	0.306	11.3	102	0.064	2.64
608	NE4.081887	0.03	0.283	15	88	0.051	2.53
609	OTT.081987	0	0.303	11.2	110	0.069	2.63
610	OTT.082087	0.01	0.288	9.7	96	0.079	2.64
611	NE4.082187	0.04	0.309	14.6	92	0.062	2.55

Table B2. (Continued)

	A	B	C	D	E	F	G
612	OTT.082187	0.01	0.247	8.2	95	0.074	2.65
613	CBF.082287	0.07	0.455	11.3	92	0.14	2.72
614	NE4.082487	0.02	0.329	11.4	95	0.074	2.46
615	OTT.082587	0.01	0.248	9.8	93	0.079	2.62
616	CBF.082687	0.05	0.687	11.9	95	0.126	2.78
617	NE4.082787	0.01	0.311	11.6	107	0.06	2.47
618	OTT.082887	0.02	0.286	10.2	101	0.106	2.66
619	OTT.082987	0.03	0.339	9.2	97	0.096	2.65
620	OTT.083187	0	0.315	10.2	101	0.1	2.65
621	OTT.090187	0.02	0.336	8.2	97	0.1	2.64
622	OTT.090287	0.01	0.278	8.9	98	0.105	2.64
623	NE4.090387	0.06	0.221	11.2	92	0.065	2.46
624	OTT.090387	0.01	0.23	9.8	98	0.098	2.64
625	CBF.090487	0.03	0.314	9.8	79	0.127	2.71
626	OTT.090487	0.01	0.291	9.5	88	0.088	2.64
627	CBF.090587	0.85	0.776	11.6	87	0.121	2.68
628	OTT.090587	0.07	0.188	10	93	0.079	2.63
629	OTT.090687	0.07	0.208	10	95	0.069	2.63
630	OTT.090887	0.05	0.25	9.8	86	0.088	2.63
631	NE4.090987	0.08	0.394	13.4	86	0.065	2.51
632	OTT.091087	0.05	0.233	10.2	92	0.095	2.62
633	NE4.091287	0.02	0.269	13.4	94	0.056	2.53
634	OTT.091287	0.01	0.262	9	86	0.083	2.67
635	CBF.091487	0.52	0.715	10.2	88	0.119	2.71
636	CBF.091587	0.36	0.712	10	87	0.134	2.7
637	NE4.091587	0.04	0.222	12.4	100	0.068	2.51
638	OTT.091587	0	0.263	10.9	89	0.112	2.64
639	CBF.091687	0.2	0.405	9.2	95	0.136	2.78
640	CBF.091787	0.39	0.622	11.6	91	0.118	2.69
641	OTT.091787	0.03	0.322	9.4	88	0.092	2.68
642	CBF.091887	0.03	0.276	9.7	92	0.141	2.73
643	NE4.091887	0	0.387	13.3	89	0.076	2.5
644	CBF.091987	0.07	0.442	9.7	97	0.132	2.73
645	CBF.092187	0.03	0.29	10.2	89	0.14	2.73
646	OTT.092287	0.07	0.325	9.7	95	0.085	2.65
647	CBF.092387	0.06	0.316	10.2	89	0.148	2.73
648	CBF.092487	0.03	0.278	9.8	84	0.139	2.72
649	OTT.092487	0.02	0.308	10.4	96	0.089	2.63
650	CBF.092587	0.04	0.204	8.8	85	0.135	2.73
651	NE4.092587	0	0.226	12.1	90	0.075	2.53
652	CBF.092887	0.05	0.255	9.6	87	0.139	2.72
653	NE4.092987	0.04	0.182	13.3	98	0.065	2.53
654	NE4.100387	0.04	0.193	12.2	90	0.055	2.49
655	CBF.100587	0.03	0.166	11.5	88	0.128	2.73
656	CBF.100687	0.05	0.301	10.6	85	0.125	2.76
657	OTT.100687	0.04	0.216	10.2	96	0.062	2.61
658	CBF.100787	0.04	0.191	10.7	82	0.125	2.74

Table B2. (Continued)

	A	B	C	D	E	F	G
659	NE4.100787	0.02	0.238	13.1	91	0.067	2.48
660	OTT.100787	0.01	0.18	10.1	92	0.051	2.61
661	OTT.100987	0	0.248	10.8	80	0.061	2.59
662	NE4.101087	0.02	0.266	14	90	0.064	2.51
663	OTT.101287	0.01	0.23	11	95	0.061	2.54
664	OTT.101387	0.01	0.231	11	84	0.053	2.52
665	CBF.101487	0.02	0.279	12.1	87	0.15	2.75
666	NE4.101487	0.02	0.255	14	94	0.086	2.49
667	CBF.101587	0.01	0.25	11.8	83	0.137	2.74
668	OTT.101587	0.01	0.192	10.6	98	0.046	2.57
669	CBF.101687	0.02	0.261	11.6	88	0.153	2.74
670	OTT.101687	0.02	0.207	11.3	91	0.05	2.56
671	NE4.101787	0.03	0.259	12.7	90	0.058	2.48
672	NE4.102087	0.04	0.229	14.2	87	0.063	2.51
673	OTT.102087	0.03	0.221	10.7	98	0.06	2.62
674	OTT.102287	0.02	0.23	10.9	97	0.057	2.62
675	OTT.102387	0.02	0.218	9.5	92	0.06	2.62
676	NE4.102487	0.04	0.247	14.9	95	0.072	2.54
677	OTT.102787	0.01	0.157	10.4	104	0.051	2.61
678	OTT.102987	0.02	0.192	10.5	103	0.04	2.54
679	NE4.110287	0.04	0.243	15.2	90	0.056	2.54
680	OTT.110387	0.02	0.158	10	107	0.037	2.55
681	OTT.110587	0.02	0.19	10.6	95	0.041	2.55
682	OTT.110987	0.02	0.182	12.5	92	0.046	2.51
683	NE4.111187	0.06	0.226	12	96	0.068	2.48
684	OTT.111187	0.02	0.177	11.9	93	0.057	2.52
685	OTT.111687	0.01	0.219	11.8	93	0.068	2.54
686	OTT.112587	0.05	0.239	13.4	97	0.06	2.56
687	NBC020188	0.03	0.208	18.1	80	0.117	2.67
688	NBC020888	0.02	0.21	17.7	81	0.115	2.68
689	NBC021588	0.03	0.233	16.2	80	0.101	2.7
690	NBC022288	0.02	0.225	17.8	80	0.104	2.61
691	NBC022988	0.02	0.183	13.4	84	0.095	2.7
692	NOS030488	0.01	0.206	11.6	85	0.086	2.7
693	NOS031188	0.01	0.208	11.1	94	0.085	2.71
694	NBC031488	0	0.258	20.5	79	0.083	2.72
695	NOS031888	0.05	0.205	11.7	92	0.086	2.7
696	NBC032188	0.01	0.246	20	81	0.089	2.7
697	NOS032588	0.03	0.242	13.1	87	0.087	2.73
698	NBC040188	0.05	0.254	19.4	82	0.102	2.67
699	NOS040188	0.03	0.26	11	85	0.088	2.74
700	OTT040688	0.04	0.305	10.6	92	0.064	2.68
701	NBC040888	0.04	0.207	16.5	81	0.093	2.67
702	NOS040888	0.04	0.191	10	82	0.113	2.69
703	OTT040888	0.02	0.256	10.9	93	0.073	2.76
704	OTT041288	0.03	0.261	11.6	90	0.069	2.67
705	OTT041488	0.02	0.217	12	97	0.059	2.64

APPENDIX C: PASTE TESTING SUMMARY AND DATA

Description of Variables in Table C1

Sample name	=	source and sampling date of the fly ash
Day No.	=	days from January 1, 1983, a reference baseline
4 hour	=	compressive strength at 4 hours (psi)
1 day	=	compressive strength at 1 day (psi)
3 day	=	compressive strength at 3 days (psi)
7 day	=	compressive strength at 7 days (psi)
14 day	=	compressive strength at 14 days (psi)
28 days	=	compressive strength at 28 days (psi)
56 days	=	compressive strength at 56 days (psi)
%exp air	=	% expansion when cured in ambient air conditions
%exp humid	=	% expansion when cured in moist air conditions
Initial set	=	first discontinuity in the penetration versus time curve (minutes)
Final set	=	arbitrarily defined as 4.5 tons per square foot (minutes)
Onset time	=	the onset point on the temperature versus time curve (minutes)
Final time	=	time required to reach the maximum temperature (minutes)
delta temp	=	difference between the final and initial temperature (degrees C)
Peak temp	=	maximum observed temperature (degrees C)
blank or •	=	missing data, due to various reasons

Table C1. Raw data for CBF, OTT and LAN paste specimens

Sample Name	Day No. (1/1/83)	4 hour	1 day	3 day	7 day	14 day	28 day	56 day	%exp air	%exp humid	Initial set	Final set	Onset time	Final time	ΔTemp	Peak Temp
OTT011385	0744	336	613	646	770	928	955	•	-0.026	-0.009	21.0	37.0		49.5	3.8	27.8
OTT022085	0782	392	614	656	823	891	1126	•	-0.034	-0.010	21.0	44.0		74.0	3.8	26.8
OTT022585	0787	448	550	584	700	890	950	•	-0.013	-0.043	12.0	18.5		57.0	7.5	29.8
OTT031585	0805	490	695	772	801	773	862	•	-0.003	-0.014	18.0	41.0		75.0	4.5	28.5
OTT032685	0816	176	282	351	360	448	578	•	-0.026	0.007	27.0	37.0		38.0	4.9	29.9
CBF040285	0823	1271	1521	•	1735	1436	•	•	•	•	•	•		•	•	•
OTT040385	0824	411	339	478	634	674	671	•	0.118	0.001	9.0	25.0		32.0	5.9	28.9
OTT041085	0831	290	376	499	481	557	665	•	-0.037	0.004	11.0	24.0		38.0	5.8	28.4
OTT041285	0833	305	315	417	591	751	689	•	-0.027	-0.007	16.0	33.0		43.0	5.1	29.1
OTT041685	0837	82	410	502	314	477	579	•	-0.029	•	36.0	107.0		82.5	3.9	27.5
OTT041985	0840	429	554	512	648	547	752	•	-0.029	-0.003	12.0	25.0		36.0	5.5	28.5
OTT042485	0845	218	393	529	490	518	467	•	0.049	-0.001	22.0	32.0		36.0	3.8	25.8
CBF042585	0846	978	1362	•	2127	2085	3089	•	-0.033	0.009	7.0	12.0		46.5	10.6	32.6
LAN043085	0851	2514	3158	2697	3742	3689	4159	•	-0.055	0.014	6.0	11.0		24.0	14.8	37.8
OTT050185	0852	223	301	•	703	906	892	•	-0.013	-0.014	31.0	40.0		44.0	3.5	26.0
OTT050285	0853	119	186	1060	2078	2186	2692	•	•	•	18.0	33.0		33.0	3.1	25.5
OTT050385	0854	151	247	479	834	785	1079	•	-0.009	•	26.0	39.0		51.0	3.2	25.7
OTT050785	0858	333	530	1002	2677	3301	3312	•	-0.080	0.026	34.0	47.0		52.0	4.4	25.9
OTT050885	0859	325	520	1016	3250	2725	2778	•	-0.029	-0.016	32.0	41.0		57.0	5.6	28.6
CBF051085	0861	1053	1153	•	1167	1192	921	•	-0.029	-0.010	9.0	16.0		53.0	8.8	29.8
OTT051085	0861	•	2375	3047	3267	4417	3990	•	•	0.024	20.0	32.0		41.0	5.4	28.9
LAN051385	0864	2682	3696	5228	4359	6250	6861	•	-0.106	0.036	2.0	8.0		•	•	•
OTT051485	0865	366	1633	3082	4721	4054	5081	•	-0.034	0.053	32.0	36.0		42.0	6.0	29.0
OTT051685	0867	357	1359	3411	4126	2417	6038	•	-0.086	0.060	25.0	30.0		52.0	4.4	25.9
OTT052085	0871	341	1438	2800	4198	5190	4875	•	•	0.013	29.0	33.0		40.0	5.3	28.8
OTT052185	0872	364	1319	3123	3157	2824	4485	•	-0.071	0.026	25.0	28.0		37.0	6.3	28.5
OTT052385	0874	385	1596	3881	3519	4648	5448	•	-0.084	0.094	22.0	27.0		29.0	6.8	29.3
CBF052485	0875	748	765	•	1010	1011	1025	•	-0.031	-0.013	12.0	17.0		31.0	7.8	28.8
OTT052885	0879	388	798	2245	2979	3017	3116	•	-0.141	0.012	26.0	31.0		36.0	6.0	30.0
OTT053085	0881	366	974	2470	2550	2507	2856	•	•	•	22.0	26.0		32.5	6.9	32.5
OTT060185	0883	396	889	1505	1886	4434	4219	•	-0.078	0.040	26.0	34.0		32.0	5.5	28.0
CBF060785	0889	707	1084	•	1230	1337	1182	•	-0.035	-0.020	11.0	17.0		25.0	9.0	31.5
OTT060785	0889	353	2017	3285	3897	5221	5273	•	-0.057	0.069	8.0	13.0		27.0	6.1	30.0
OTT061085	0892	247	330	742	1394	2718	2486	•	-0.050	0.013	24.0	34.0		38.0	4.1	26.1
OTT061285	0894	208	•	1409	1761	1875	2870	•	-0.046	0.000	23.0	28.0		30.0	5.0	27.0

Table C1. (Continued)

Sample Name	Day No. (1/1/83)	4 hour	1 day	3 day	7 day	14 day	28 day	56 day	%exp air	%exp humid	Initial set	Final set	Onset time	Final time	ΔTemp	Peak Temp
CBF061385	0895	693	1154	•	1551	1584	1660	•	-0.040	-0.031	•	•		•	•	•
OTT061785	0899	305	1462	2630	3135	3530	3900	•	-0.055	0.036	11.0	13.0		36.0	6.0	28.5
LAN061985	0901	2239	3262	3477	4281	3704	4949	•	-0.047	0.015	6.0	9.0		24.0	14.8	38.3
OTT061985	0901	302	984	2172	3102	2577	4300	•	-0.048	0.021	8.0	12.0		41.0	5.5	28.5
CBF062585	0907	853	1467	•	5669	3599	6681	•	-0.045	0.009	10.0	14.0		25.6	8.5	31.5
LAN062585	0907	1973	3571	2994	3472	3941	4700	•	-0.039	0.029	•	•		•	•	•
OTT062585	0907	414	656	1107	1181	1453	1667	•	-0.046	-0.010	26.0	42.0		58.0	6.5	28.5
OTT062885	0910	430	419	957	1760	3425	3221	•	•	•	11.0	16.0		20.0	5.9	28.9
CBF070185	0913	530	580	•	820	•	1209	•	-0.009	-0.006	5.0	14.0		•	•	•
OTT070185	0913	407	523	863	1527	2657	2944	•	-0.017	0.016	10.0	14.0		22.0	6.0	27.7
LAN070685	0918	1357	2417	2520	2457	2795	3312	•	-0.030	-0.001	9.0	11.0		•	•	•
CBF070885	0920	805	1208	•	1659	1689	1940	•	-0.029	-0.017	•	•		•	•	•
OTT070885	0920	613	1065	1863	2770	2827	3592	•	-0.037	0.001	10.0	14.0		56.0	7.0	29.0
OTT071285	0924	491	825	1169	1135	1377	1709	•	-0.047	-0.021	36.0	57.0		67.0	6.3	28.8
LAN071385	0925	2206	2662	3490	3934	4247	5623	•	-0.046	0.003	•	•		17.0	14.3	38.3
OTT071985	0931	538	798	1170	1310	1449	1105	•	-0.040	-0.021	•	•		40.0	7.2	29.8
LAN072085	0932	2246	4106	4959	5316	5496	5752	•	-0.052	0.096	•	•		•	•	•
CBF072385	0935	1034	1173	•	1285	1182	1724	•	-0.026	-0.020	7.0	11.0		44.0	8.5	31.5
OTT072685	0938	460	674	958	985	911	1316	•	-0.033	-0.010	31.0	51.0		65.0	4.5	26.5
LAN072785	0939	1843	3002	3474	3808	3449	4909	•	-0.050	0.021	6.0	9.0		22.0	17.8	40.8
CBF072985	0941	1193	1142	•	1379	1559	1426	•	-0.040	-0.028	11.0	19.0		44.0	7.8	28.3
OTT080185	0944	381	469	670	798	973	1120	•	-0.038	-0.008	17.0	21.0		30.0	5.6	30.8
LAN080285	0945	2300	4235	3584	5229	6737	7130	•	-0.104	0.033	4.0	6.0		•	•	•
OTT080585	0948	357	643	905	711	952	1106	•	-0.038	-0.017	31.0	50.0		60.0	6.6	28.1
CBF080685	0949	1146	1255	1473	1519	1625	1878	•	-0.063	-0.019	4.0	11.0		•	•	•
OTT080985	0952	390	690	1153	1244	1288	1709	•	-0.056	-0.021	24.0	27.0		40.0	5.0	28.3
LAN081085	0953	1297	1506	1824	2373	1771	1940	•	•	•	7.0	11.0		40.0	12.3	34.3
OTT081285	0955	270	718	1201	1424	1211	1875	•	-0.040	-0.005	31.0	39.0		43.0	5.3	26.5
OTT081585	0958	348	342	692	896	990	1041	•	-0.054	-0.006	19.0	25.0		35.0	6.3	28.8
OTT082085	0963	428	581	701	1047	1003	1020	•	-0.033	-0.005	•	•		37.0	5.0	29.0
LAN082485	0967	2396	3462	4123	4567	5196	6204	•	-0.088	0.050	5.0	9.5		•	•	•
OTT082685	0969	432	•	1249	1679	1189	1894	•	-0.043	-0.019	21.0	32.0		48.0	7.4	29.4
CBF082785	0970	734	1232	1728	2030	2221	2502	•	-0.049	-0.014	10.0	16.0		29.0	11.0	34.0
OTT083085	0973	442	538	693	1019	965	1021	•	-0.067	-0.017	25.0	42.0		63.5	6.5	27.5
OTT090585	0979	577	•	1062	1048	1539	1438	•	-0.041	-0.020	26.0	43.0		70.0	6.8	28.5

Table C1. (Continued)

Sample Name	Day No. (1/1/83)	4 hour	1 day	3 day	7 day	14 day	28 day	56 day	%exp air	%exp humid	Initial set	Final set	Onset time	Final time	ΔTemp	Peak Temp
CBF090685	0980	475	759	1128	1298	1293	1910	•	-0.041	-0.021	14.0	26.0		42.0	8.5	30.5
CBF091185	0985	1122	1538	1484	2083	3300	4716	•	-0.063	-0.004	12.0	17.0		36.0	10.5	32.5
OTT091285	0986	574	645	1038	1061	1229	1493	•	-0.062	-0.008	20.0	28.0		•	•	•
CBF091685	0990	1057	1407	1737	2324	3883	4542	•	-0.061	0.003	12.0	18.0		48.0	10.5	32.5
CBF092085	0994	1114	964	1240	1388	1893	2327	•	-0.030	0.009	10.0	20.0		58.0	10.3	31.3
OTT092085	0994	492	•	900	789	1156	1041	•	-0.034	-0.008	16.0	23.0		31.0	8.3	30.3
LAN092185	0995	1684	2251	2784	3814	3612	4391	•	-0.043	0.015	10.0	13.0		26.0	12.5	34.5
OTT092585	0999	333	•	672	698	1259	1328	•	-0.060	-0.023	23.0	39.0		51.0	5.0	27.8
CBF093085	1004	802	1118	•	1442	1434	2308	•	-0.030	-0.023	13.0	19.0		37.0	10.5	32.5
OTT100485	1008	302	•	808	704	1025	1077	•	-0.048	-0.018	18.0	52.0		59.0	4.0	26.0
OTT100885	1012	494	538	689	1205	1382	1128	•	-0.053	-0.015	15.0	42.0		71.0	4.5	26.6
CBF101185	1015	870	1062	•	1477	2086	3239	•	-0.046	-0.018	16.0	21.0		34.0	12.0	34.0
LAN101185	1015	78	316	706	792	772	787	•	•	•	7.0	50.0		28.0	4.3	25.8
OTT101285	1016	356	567	641	1013	1332	1067	•	-0.053	-0.033	19.0	29.0		55.0	5.0	26.0
OTT101785	1021	301	416	699	898	860	1047	•	-0.062	-0.012	14.0	27.0		55.0	3.9	24.9
OTT102285	1026	232	387	791	829	966	1224	•	-0.050	-0.014	19.0	31.0		48.0	4.9	27.4
OTT102885	1032	226	280	586	642	805	920	•	-0.020	0.011	13.0	52.0		22.0	5.5	28.5
OTT110485	1039	216	342	638	792	867	•	•	-0.034	-0.016	14.0	29.0		29.0	5.0	27.4
OTT112285	1057	205	416	284	546	495	607	•	-0.007	-0.003	12.0	23.0		46.0	2.8	25.3
CBF021986	1146	1555	1878	2981	4330	5325	5918	•	0.008	0.309	6.0	8.0		17.0	17.0	41.5
LAN030786	1162	1118	1374	•	2942	2526	2731	•	•	0.019	8.0	15.0		•	•	•
OTT031386	1168	217	230	•	438	566	605	•	-0.021	-0.015	19.0	26.0		30.5	5.8	30.8
OTT040386	1189	215	296	•	487	597	595	•	-0.023	-0.014	20.0	26.0		43.0	5.6	30.1
LAN040786	1193	1596	1622	•	2574	3956	4050	•	-0.018	0.066	•	•		20.5	16.6	39.6
OTT041286	1198	210	329	317	357	363	525	•	-0.013	0.005	24.0	42.0		39.0	6.6	28.6
CBF041886	1204	887	1567	3602	4872	4699	5361	•	-0.013	0.134	5.0	7.0		13.0	11.3	35.8
OTT042186	1207	285	539	480	548	653	756	•	-0.017	-0.005	13.0	23.0		40.0	7.5	29.5
LAN042586	1211	1647	1727	•	2418	3543	3959	•	-0.027	-0.013	•	•		56.5	12.1	35.1
OTT042886	1214	278	326	339	376	510	439	•	-0.011	-0.001	12.0	22.0		44.0	5.8	28.3
CBF043086	1216	914	1265	1586	2901	4829	5238	•	-0.024	0.112	7.0	8.0		17.0	12.5	36.5
OTT050186	1217	412	715	880	959	1056	1229	•	-0.034	-0.021	24.0	35.0		58.0	6.9	28.9
CBF050686	1222	1130	1709	•	2528	3438	3838	•	-0.020	0.039	8.0	10.0		17.0	16.0	39.0
OTT050786	1223	273	449	•	767	969	1176	•	-0.037	-0.016	28.0	34.0		44.0	4.4	26.4
CBF050986	1225	1715	2154	•	3626	5162	3732	•	-0.032	0.075	7.0	9.0		19.0	17.8	40.8
OTT050986	1225	201	598	•	2497	2296	2728	•	-0.024	0.017	14.0	19.0		22.0	5.0	27.5

Table C1. (Continued)

Sample Name	Day No. (1/1/83)	4 hour	1 day	3 day	7 day	14 day	28 day	56 day	%exp air	%exp humid	Initial set	Final set	Onset time	Final time	ΔTemp	Peak Temp
OTT051386	1229	283	605	•	1168	1244	1791	•	-0.033	-0.007	23.0	27.0		34.0	5.8	26.8
OTT051686	1232	203	423	•	1897	1723	2026	•	-0.022	0.003	13.0	19.0		23.0	5.3	28.3
CBF051986	1235	2057	2597	•	3262	4625	4908	•	-0.043	0.039	7.0	9.0		19.0	14.8	36.8
LAN052086	1236	1979	2368	•	3916	5018	4987	•	-0.039	0.050	5.0	7.0		25.0	16.0	38.0
OTT052186	1237	176	321	751	1088	1357	1655	•	-0.024	-0.002	17.0	25.0		26.0	4.4	27.4
OTT052386	1239	87	191	514	966	1342	1213	•	-0.015	0.001	27.0	39.0		30.0	3.5	27.0
CBF052786	1243	1245	2624	•	4158	5036	5943	•	-0.066	0.027	8.0	10.0		19.0	10.5	32.0
OTT052886	1244	212	348	775	1481	1483	2061	•	-0.025	-0.002	17.0	22.0		23.5	4.5	27.5
CBF053086	1246	998	1929	1524	2958	3943	4487	•	-0.035	0.097	•	•		19.5	13.2	35.2
OTT053186	1247	274	415	•	881	1058	1333	•	-0.046	-0.019	20.0	29.0		38.0	4.3	28.3
OTT060486	1251	229	360	414	569	741	858	•	-0.035	-0.005	17.0	23.0		24.0	5.0	29.0
CBF060586	1252	937	1398	•	4139	4737	6086	•	-0.032	0.173	8.0	9.0		17.0	12.3	35.3
OTT060686	1253	241	325	426	689	536	827	•	-0.026	-0.013	28.0	36.0		41.0	5.0	29.0
CBF060986	1256	998	1354	•	4239	4768	6056	•	-0.047	0.107	10.0	11.0		20.0	10.0	33.0
OTT061286	1259	258	319	761	1759	1783	1896	•	-0.011	-0.009	11.0	28.0		37.0	3.8	26.8
OTT061386	1260	132	266	242	417	442	521	•	•	-0.001	34.0	77.0		75.0	3.8	27.8
CBF061686	1263	867	1392	•	2035	3569	4991	•	-0.047	0.026	9.0	12.0		20.0	11.8	37.8
OTT061786	1264	216	262	•	395	510	587	•	-0.013	-0.004	13.0	25.0		37.0	4.0	27.0
OTT061986	1266	351	426	400	571	644	664	•	-0.021	-0.006	15.0	26.0		35.0	6.3	32.3
CBF062386	1270	1065	1563	1952	3124	3742	3717	•	-0.082	0.004	8.0	10.0		19.0	9.5	34.5
OTT062386	1270	357	525	575	698	1032	812	•	-0.031	-0.021	21.0	33.0		48.0	6.0	31.5
OTT062486	1271	342	557	688	1035	1082	1328	•	•	-0.003	22.0	37.0		48.0	6.3	31.3
CBF062686	1273	811	1447	1591	1712	2767	3435	•	-0.089	-0.004	8.0	10.0		20.0	9.8	34.3
OTT062686	1273	357	490	697	1034	1172	1331	•	-0.035	-0.029	22.0	34.0		52.0	6.5	30.5
CBF070186	1278	1174	1701	1821	2452	2809	3713	•	-0.092	0.038	7.0	10.0		18.0	8.8	33.8
OTT070186	1278	398	751	696	991	908	1296	•	-0.031	-0.022	22.0	40.0		58.0	4.5	29.5
LAN070286	1279	2010	4123	4048	5340	4451	5596	•	-0.067	0.042	6.0	7.0		18.0	16.5	42.0
OTT070386	1280	349	605	619	899	796	744	•	-0.030	-0.016	18.0	29.0		48.0	6.4	31.4
OTT070786	1284	462	524	569	608	662	625	•	•	-0.008	12.0	27.0		46.0	5.9	30.9
CBF071086	1287	1057	1845	•	3443	3809	4897	3938	-0.068	-0.025	11.5	12.5		21.0	8.5	33.5
OTT071086	1287	329	465	440	571	677	520	•	-0.021	-0.008	12.0	22.0		39.0	5.1	29.1
LAN071486	1291	1707	2696	3106	3238	3097	5576	•	-0.064	0.095	7.0	8.0		18.0	15.8	40.8
OTT071686	1293	289	384	472	574	629	546	•	-0.025	-0.010	18.0	27.0		47.0	4.8	29.8
CBF071886	1295	1013	1624	•	2299	3305	3951	4706	-0.099	0.014	16.5	19.0		31.0	9.2	33.2
OTT072186	1298	315	455	482	535	685	928	•	-0.014	0.003	14.0	22.0		32.0	6.3	31.3

Table C1. (Continued)

Sample Name	Day No. (1/1/83)	4 hour	1 day	3 day	7 day	14 day	28 day	56 day	%exp air	%exp humid	Initial set	Final set	Onset time	Final time	ΔTemp	Peak Temp
CBF072486	1301	1325	1452	•	2308	2649	5526	6398	-0.081	0.048	16.7	20.2		33.5	14.0	39.0
OTT072486	1301	•	450	550	732	682	695	•	-0.043	-0.003	15.0	21.0		36.0	5.8	28.8
LAN073086	1307	•	•	•	•	•	7536	•	-0.111	0.072	10.0	13.0		22.0	16.6	38.6
OTT073186	1308	340	453	•	642	770	717	•	•	•	23.0	29.0		•	•	•
CBF080186	1309	1411	1642	•	2357	3709	4877	5543	-0.076	0.087	18.5	21.5		31.0	14.7	39.7
OTT080486	1312	•	•	•	703	707	800	•	-0.046	-0.033	29.0	37.0		•	•	•
OTT080786	1315	300	•	•	632	772	•	•	•	•	37.0	50.0		61.0	3.6	26.6
CBF080886	1316	1788	1808	•	2862	4220	4819	5642	-0.034	0.090	18.0	20.5		35.5	16.2	41.7
LAN081186	1319	•	3199	•	•	•	6935	•	-0.099	0.099	13.0	15.0		22.5	15.9	37.9
OTT081186	1319	189	588	•	644	951	•	•	•	•	32.0	47.0		48.0	4.0	26.0
CBF081286	1320	1802	1937	•	2866	5046	5631	6933	-0.030	0.075	16.5	18.5		31.0	16.1	40.6
OTT081486	1322	•	374	•	575	430	571	•	-0.040	-0.004	50.0	60.0		69.0	2.5	27.0
LAN081886	1326	1743	2482	•	3115	4254	5035	4207	-0.074	0.074	9.5	12.0		25.0	14.9	39.9
OTT081886	1326	•	400	•	490	542	604	•	•	-0.007	44.0	55.0		59.0	2.2	25.7
CBF081986	1327	1913	2328	•	2863	4534	6406	5805	•	•	16.0	18.5		32.0	13.5	39.5
CBF082286	1330	1930	2490	•	2881	3957	4301	5225	•	•	13.0	14.5		23.0	15.0	41.0
OTT082286	1330	•	367	•	505	785	827	•	-0.040	-0.003	31.0	38.0		•	•	•
LAN082586	1333	2401	3155	•	5354	4778	6551	5753	-0.095	0.073	13.0	17.0		27.0	16.5	41.0
CBF082686	1334	1671	1752	•	2455	•	3811	4530	-0.043	0.032	15.0	16.0		29.0	11.5	36.5
OTT082886	1336	•	312	•	588	742	727	•	•	•	34.0	41.0		52.5	3.6	27.6
CBF082986	1337	1517	1653	•	1974	•	3708	4388	-0.055	0.054	13.0	15.0		27.0	12.8	38.8
OTT090286	1341	•	481	•	851	809	906	•	-0.033	-0.002	17.0	21.0		30.0	5.9	27.9
CBF090486	1343	1138	1436	•	1900	1992	3686	4509	-0.042	0.109	•	11.0		31.0	11.0	36.5
CBF090586	1344	984	1011	•	1671	1597	2256	4177	-0.052	•	11.5	13.8		31.0	9.8	34.8
CBF090686	1345	812	888	•	1564	1761	2159	3402	-0.076	0.079	18.8	20.8		31.0	10.5	35.5
OTT090686	1345	•	300	•	487	516	631	•	•	0.002	25.0	32.0		42.5	4.5	26.5
CBF090886	1347	962	1066	•	1768	1858	2825	3626	-0.043	0.015	13.0	15.0		41.0	8.2	33.7
LAN090886	1347	1567	2073	•	2899	3761	4253	4319	-0.084	-0.010	8.0	9.0		25.0	15.0	41.0
OTT090986	1348	•	501	•	603	568	720	•	-0.040	0.009	25.0	33.0		44.0	5.3	27.3
CBF091086	1349	1068	1617	•	1985	2325	4001	3666	•	0.057	11.7	13.2		29.0	11.7	37.7
OTT091586	1354	•	469	•	699	838	885	•	-0.051	-0.012	29.0	36.0		40.0	4.0	26.0
CBF091686	1355	1143	•	•	2221	3193	4010	4332	-0.051	0.056	10.5	12.5		28.0	10.2	36.7
OTT091986	1358	•	540	•	688	888	737	•	-0.029	-0.003	23.0	29.0		38.0	5.1	28.1
LAN092386	1362	1787	2249	•	3445	4657	4461	4827	-0.111	0.158	5.6	7.0		35.0	12.0	37.0
OTT092486	1363	•	423	•	614	890	775	•	-0.035	0.000	22.0	30.0		32.0	5.6	27.6

Table C1. (Continued)

Sample Name	Day No. (1/1/83)	4 hour	1 day	3 day	7 day	14 day	28 day	56 day	%exp air	%exp humid	Initial set	Final set	Onset time	Final time	ΔTemp	Peak Temp
LAN092986	1368	1780	1525	•	2001	2828	3197	3936	-0.124	-0.007	5.0	6.5		44.0	12.7	37.7
OTT092986	1368	•	512	•	599	605	905	•	-0.028	0.001	20.0	26.0		•	•	•
LAN100686	1375	2319	3159	•	4063	5429	4565	5552	-0.109	0.073	10.7	12.2		23.0	19.0	43.5
OTT100686	1375	•	349	•	•	608	722	•	-0.030	0.003	20.0	26.0		22.5	6.4	27.4
OTT101086	1379	•	463	•	560	717	747	•	-0.037	0.025	36.0	52.0		70.0	3.5	24.5
LAN101586	1384	1890	2265	•	2773	4061	4761	4749	-0.070	0.089	12.3	13.3		21.0	17.3	42.3
OTT101786	1386	•	289	•	452	464	504	•	-0.026	0.010	33.0	43.0		33.0	5.0	27.0
LAN102286	1391	1575	2281	•	3228	3402	4042	3228	-0.084	0.028	10.0	12.0		33.0	11.8	36.8
OTT102386	1392	•	509	•	755	603	895	•	-0.015	0.006	18.0	25.0		29.0	4.3	26.3
LAN102786	1396	1929	2515	•	4067	4397	5127	4496	-0.051	0.043	8.0	9.5		25.0	15.3	42.3
OTT103186	1400	•	489	•	641	640	962	•	-0.013	-0.003	15.0	22.0		•	•	•
LAN110386	1403	1749	1965	•	3534	4674	4800	4344	-0.069	0.094	8.0	9.0		22.0	14.2	40.7
OTT111086	1410	•	467	•	2219	•	2688	•	•	0.133	19.0	24.0		29.0	2.8	25.8
CBF120886	1438	1284	1602	•	3514	4732	6102	4896	-0.053	0.060	6.0	7.2		29.0	10.7	36.7
OTT121586	1445	•	2467	•	2827	•	4526	•	-0.028	0.123	12.0	14.0		20.0	4.7	27.2
OTT011587	1476	402	605		692	899	696	637	-0.038	-0.004	12.0	17.0		34.0	4.8	30.8
OTT021587	1507	293	452		671	644	639	753	-0.037	-0.014	15.0	22.0		39.0	4.8	29.8
OTT031687	1536	305	541		645	659	560	515	-0.029	-0.009	13.0	18.0		34.0	4.6	29.6
OTT040287	1553	298	416		489	538	359	459		0.007	10.0	17.0		36.0	3.7	28.7
OTT040887	1559	306	361		560	553	422	485	-0.025	0.014	9.0	19.0		32.0	5.5	30.5
OTT041587	1566	250	542		721	709	646	514	-0.045	0.008	9.0	36.0		17.0	3.0	27.0
OTT041787	1568	301	546		691	658	690	625	-0.041	0.019	10.0	31.0		33.0	4.8	28.8
LAN042087	1571	2610	4232		5587	5164	6638	6681	-0.060	0.007	6.0	8.5				
OTT042187	1572	63	112		132	173	148	156			21.0	32.0		38.0	5.0	30.0
OTT042487	1575	423	557		689	592	558	643	-0.037	0.019	8.0	16.0		28.0	5.5	28.5
OTT042887	1579	505	674		710	844	676	943	-0.032	0.102	8.0	24.0		29.0	4.8	29.8
LAN050487	1585	2824	3471		5474	6026	5827	7256	-0.150	0.015	8.0	10.0				
OTT050587	1586	306	425		575	465	467	653		0.026	14.0	27.0		24.0	4.5	28.5
OTT050887	1589	387	497		791	1009	862	1021	-0.042	-0.012	17.0	29.0		36.0	4.4	27.4
LAN051087	1591	2494	3823		6043	6555	6925	8323	-0.167	0.010	6.0	9.0				
LAN052687	1607	2489	4374		5524	6562	7600	9335	-0.156	0.009	9.0	11.5	26.0	31.0	10.2	33.2
LAN053187	1612	2423	4741		6078	8196	7348	8611	-0.141	0.019	9.5	12.0				
OTT060987	1621	152	202		321	343	479	406		0.003	14.0	22.0		19.0	2.8	25.8
OTT061187	1623	101	148		197	220	365	352	-0.017					11.0	1.0	25.0
OTT061587	1627	113	251		354	347	512	375	-0.021	0.004				25.0	3.2	26.7

Table C1. (Continued)

Sample Name	Day No. (1/1/83)	4 hour	1 day	3 day	7 day	14 day	28 day	56 day	%exp air	%exp humid	Initial set	Final set	Onset time	Final time	ΔTemp	Peak Temp
OTT061787	1629	284	293		372	374	380	305		0.007	16.0	22.0		30.0	3.9	27.4
OTT062287	1634	246	370		443	412	477	349	-0.033	0.000	15.0	21.0		35.0	3.2	27.2
OTT062487	1636	310	511		785	736	1033	•	-0.085	-0.005	20.0	28.0		37.0	4.9	28.4
OTT062787	1639	235	518		565	561	625	•	-0.047	-0.013	27.0	33.0		44.0	3.3	27.3
LAN062987	1641	2561	3977		6533	6712	8180	7889	-0.135	-0.004	17.0	20.5	29.0	30.0	12.2	34.2
LAN070287	1644	2336	5074		6869	7801	8140	8884	-0.149	0.035	12.5	14.5	18.0	19.0	14.5	35.5
OTT070287	1644	269	370		694	830	902	1169	-0.070	-0.016	28.0	33.0		41.0	3.3	28.3
OGS070887	1650	280	423		914	970	1395	1483	-0.061	-0.017	23.0	27.0		25.0	4.5	29.5
LGS070987	1651	318	988		2517	2538	4289	3110			9.0	13.0	15.0	39.0	2.5	
LAN071387	1655	2415	3440		6400	6709	7802	8595	-0.156	0.041	14.5	16.0	15.0	17.0	10.5	32.5
LGS071587	1657	931	1417		3259	4108	3410	4196	-0.120	0.087	7.0	9.0	21.0	35.0	6.5	31.5
OGS071587	1657	399	793		1087	1571	1408	2076		-0.011	24.0	29.0		34.0	4.3	28.3
LGS072187	1663	476	1139		3737	3853	3202	3812	-0.122	0.056	10.0	12.0	23.0	35.0	4.8	29.8
OGS072287	1664	566	628		895	919	1112	1256	-0.046	0.009	10.0	14.0	20.0	26.0	6.8	29.8
LAN072887	1670	1587	2123		4687	4490	5484	6705	-0.140	0.089	13.0	14.0	17.0	20.0	10.5	33.0
LGS072987	1671	702	1796		2955	4094	3849	3900		0.098	7.0	10.0	13.0	37.0	4.0	28.5
OGS072987	1671	305	490		818	876	1014	1211	-0.036	-0.020	11.0	15.0		23.0	4.3	28.3
LAN080387	1676	2013	3074		5245	4962	5137	9056	-0.129	0.073	3.5	4.5	13.0	15.0	10.8	33.8
LGS080487	1677	624	1554		2105	4322	3897	4094		0.094	8.0	12.0	15.0	36.0	3.0	26.0
OGS080587	1678	364	512		778	893	806	972	-0.038	0.002	19.0	38.0		24.0	5.0	29.0
LGS081287	1685	232	368		1267	2348	2241	2364	-0.055	0.070	7.0	10.0	9.0	20.0	2.8	27.8
OGS081287	1685	249	363		520	590	696	796			43.0	57.0	40.0	48.0	3.8	26.8
OGS081987	1692	290	394		483	497	704	743	-0.042		22.0	36.0		30.0	4.0	28.0
LGS082087	1693	327	730		1747	2578	3459	2978		0.150	4.5	5.5	7.0	19.0	4.5	28.5
LGS082687	1699	648	1392		3088	3928	4458	4088		0.073	7.0	10.0	13.0	34.0	6.3	31.3
OGS082687	1699	550	723		886	1004	984	1161	-0.076	-0.011	12.0	30.0		46.0	3.8	26.8
OGS090287	1706	498	558		730	770	777	1076	-0.074	0.002	9.0	21.0	26.0	38.0	6.5	27.5
LGS090487	1708	531	1430		3523	3596	2999	4121		0.085	7.0	10.0	14.0	30.0	4.3	28.3
OGS090987	1713	434	577		666	691	809	1041			11.0	16.0		28.0	6.5	29.5
LGS091087	1714	524	755		2720	3653	3369	2862		0.010	10.0	14.0	12.0	26.0	3.3	26.8
OGS091487	1718	390	496		600	788	902	956	-0.064	0.009	10.0	16.0	23.0	34.0	6.2	27.2
LGS091687	1720	661	822		3515	3844	3545	4408	-0.156	0.028	12.0	17.0	13.0	35.0	2.8	29.8
OGS091687	1720	115	247		384	406	412	435			19.0	27.0	34.0	47.0	3.5	26.5
OGS091887	1722	79	158		238	258	236	229			33.0	51.0	31.0	52.0	2.5	24.2
LGS092087	1724	671	1001		3540	4499	3510	4066	-0.186	0.018	8.0	11.0	10.0	23.0	6.3	30.3

Table C1. (Continued)

Sample Name	Day No. (1/1/83)	4 hour	1 day	3 day	7 day	14 day	28 day	56 day	%exp air	%exp humid	Initial set	Final set	Onset time	Final time	ΔTemp	Peak Temp
OGS092187	1725	316	501		735	863	675	1095	-0.057	0.010	15.0	21.0	36.0	56.0	1.8	24.8
OGS092387	1727	306	356		552	597	692	845		-0.001	14.0	23.0	18.0	27.0	7.3	30.2
LGS092487	1728	936	1746		3893	4337	3362	4368		0.037	8.0	11.0	10.0	27.0	7.8	31.3
OGS092587	1729	328	408		670	762	738	807	-0.037	-0.007	10.0	14.0	21.0	26.0	4.5	27.5
LGS092687	1730	829	1625		3458	3559	4701	4832		0.000	12.0	19.0	14.0	47.0	5.2	29.2
OGS100687	1740	33	173		347	265	240	532	-0.026	-0.005	94.0	178.0	9.0	15.0	0.5	22.5
OGS100787	1741										97.0	198.0	6.0	12.0	0.3	22.8
OGS100887	1742	237	360		613	1577	2277	2591	-0.034		23.0	27.0	19.0	42.0	3.0	26.0
OGS100987	1743	199	298		1793	1996	2169	3342	-0.029		35.0	41.0	9.0	20.0	0.5	23.5
OGS101287	1746	207	621		2273	2508	2030	2991	-0.035		11.0	15.0	7.0	14.0	1.3	24.8
LAN102387	1757	1403	1560		2267	2563	2790	3772	-0.079	0.069	9.5	10.5	24.0	28.0	12.0	34.5
LGS103087	1764	280	1103		2242	2834	3239	3752	-0.061	0.128	6.0	8.0	9.0	18.0	3.8	27.3
LGS110187	1766	300	646		1985	1593	2944	3288		0.088	7.0	11.0	14.0	19.0	3.5	26.5
LGS110387	1768	259	862		2767	1940	3526	3853		0.111	7.0	10.0	15.0	20.0	3.4	26.4
LGS110587	1770	230	832		2738	1817	3253	3471		0.107	9.0	14.0	25.0	21.0	2.3	25.3
OTT111687	1781	218	417		1786	1600	1930	2229		0.050	11.0			22.0	3.1	27.1
OTT112587	1790		357		514	571	790	1193			49.0			23.0	0.6	24.6
OTT121587	1810	275	420		2006	2731	3382	4022	-0.032	0.006	12.0	19.0		23.0	4.3	27.3
LAN121887	1813	1402	1282		1910	2213	2272	3024	-0.066	0.027	4.0	5.5	28.0	36.0	9.0	31.5
Samples	264	239	253	106	260	256	259	98	216	236	250	249	40	242	242	241
Min	0744	33	112	242	132	173	148	156	-0.186	-0.043	2.0	4.5	6.0	11.0	0.3	22.5
Max	1813	2824	5074	5228	6869	8196	8180	9335	0.118	0.309	97.0	198.0	40.0	82.5	19.0	43.5
Range	1069	2791	4962	4986	6737	8023	8032	9179	0.304	0.352	95.0	193.5	34.0	71.5	18.7	21.0
Average	1273	782	1152	1457	1926	2211	2620	3329	-0.050	0.021	16.5	24.8	18.0	35.1	7.2	30.6
St Deviation	0303	687	1009	1164	1501	1742	2024	2409	0.037	0.046	11.4	20.1	8.5	13.8	4.1	4.6

Correlations Between Physical Properties

The results of correlation studies using the fly ash paste data from Council Bluffs, Lansing and Ottumwa power plants are shown in Tables C2, C3 and C4, respectively. Abbreviations for the variables were as follows:

H4	=	4 hour compressive strength
D1	=	1 day compressive strength
D7	=	7 day compressive strength
D14	=	14 day compressive strength
D 28	=	28 day compressive strength
D56	=	56 day compressive strength
ACE	=	air cured expansion
HCE	=	humid cured expansion
IS	=	initial set time
FS	=	final set time
PKT	=	peak temperature
TIM	=	time required to reach peak temperature
DT	=	temperature rise (final temp - initial temp)

Linear correlation coefficients were generated by using the combined 1985 and 1986 paste test results from each of the individual power plants. The tables also list the significance probability of the correlation and the number of observations that were used in calculating the statistics. For example, in Table C2 (the Council Bluffs paste data), the Pearson correlation coefficient, r, between the 4-hour compressive strength (H4) and the one-day compressive strength (D1) was 0.79516. The number directly below the correlation coefficient (0.0001 in this instance) is the significance probability of the correlation. This value indicates that the linear correlation between H4 and D1 was significant (i.e., we can reject the null hypothesis that no linear relationship (r=0) exists between H4 and D1). We have arbitrarily adopted a 99% confidence interval for accepting or rejecting potential correlations, this corresponds to a significance probability value of 0.005 or less. The integer directly below the significance probability of the correlation value denotes the number of samples used in the statistical calculations (49 observations in this instance). Please note that all of the correlation matrices are symmetric about their main diagonals.

Table C2. Correlation matrix for CBF paste specimens (1985-86)

PEARSON CORRELATION COEFFICIENTS / PROB > |R| UNDER HO:RHO=0 / NUMBER OF OBSERVATIONS

	H4	D1	D7	D14	D28	D56	ACE	HCE	IS	FS	PKT	TIM	DT
H4	1.00000 0.0000 50	0.79516 0.0001 49	0.33773 0.0165 50	0.55686 0.0001 47	0.47624 0.0005 49	0.72055 0.0011 17	-0.01532 0.9195 46	0.38985 0.0074 46	0.19534 0.1985 45	-0.08591 0.5703 46	0.67845 0.0001 45	-0.12637 0.4081 45	0.54653 0.0001 45
D1	0.79516 0.0001 49	1.00000 0.0000 49	0.60390 0.0001 49	0.73714 0.0001 46	0.66452 0.0001 48	0.53826 0.0315 16	-0.15497 0.3094 45	0.36935 0.0126 45	-0.00789 0.9595 44	-0.33781 0.0232 45	0.57799 0.0001 44	-0.48691 0.0008 44	0.50379 0.0005 44
D7	0.33773 0.0165 50	0.60390 0.0001 49	1.00000 0.0000 50	0.83652 0.0001 47	0.84010 0.0001 49	0.46224 0.0617 17	0.05708 0.7063 46	0.58252 0.0001 46	-0.22540 0.1366 45	-0.50103 0.0004 46	0.32929 0.0272 45	-0.61288 0.0001 45	0.30857 0.0392 45
D14	0.55686 0.0001 47	0.73714 0.0001 46	0.83652 0.0001 47	1.00000 0.0000 47	0.88576 0.0001 46	0.67112 0.0062 15	0.05656 0.7187 43	0.64965 0.0001 43	-0.16050 0.3099 42	-0.45329 0.0023 43	0.60689 0.0001 43	-0.61705 0.0001 43	0.50726 0.0005 43
D28	0.47624 0.0005 49	0.66452 0.0001 48	0.84010 0.0001 49	0.88576 0.0001 46	1.00000 0.0000 49	0.74128 0.0007 17	-0.11987 0.4275 46	0.60595 0.0001 46	0.03792 0.8047 45	-0.32342 0.0283 46	0.54105 0.0001 45	-0.52273 0.0002 45	0.35833 0.0156 45
D56	0.72055 0.0011 17	0.53826 0.0315 16	0.46224 0.0617 17	0.67112 0.0062 15	0.74128 0.0007 17	1.00000 0.0000 17	0.12762 0.6637 14	0.32807 0.2522 14	0.36417 0.1655 16	0.41111 0.1011 17	0.71440 0.0013 17	0.11288 0.6662 17	0.41591 0.0968 17
ACE	-0.01532 0.9195 46	-0.15497 0.3094 45	0.05708 0.7063 46	0.05656 0.7187 43	-0.11987 0.4275 46	0.12762 0.6637 14	1.00000 0.0000 46	0.30602 0.0409 45	-0.34019 0.0275 42	-0.17818 0.2530 43	0.10052 0.5265 42	0.05166 0.7453 42	0.41461 0.0063 42
HCE	0.38985 0.0074 46	0.36935 0.0125 45	0.58252 0.0001 46	0.64965 0.0001 43	0.60595 0.0001 46	0.32807 0.2522 14	0.30602 0.0409 45	1.00000 0.0000 46	-0.10238 0.5188 42	-0.39037 0.0097 43	0.63974 0.0001 42	-0.47459 0.0015 42	0.46323 0.0020 42
IS	0.19534 0.1985 45	-0.00789 0.9595 44	-0.22540 0.1366 45	-0.16050 0.3099 42	0.03792 0.8047 45	0.36417 0.1655 16	-0.34019 0.0275 42	-0.10238 0.5188 42	1.00000 0.0000 45	0.82370 0.0001 45	0.18146 0.2442 43	0.33200 0.0296 43	-0.10450 0.5048 43
FS	-0.08591 0.5703 46	-0.33781 0.0232 45	-0.50103 0.0004 46	-0.45329 0.0023 43	-0.32342 0.0283 46	0.41111 0.1011 17	-0.17818 0.2530 43	-0.39037 0.0097 43	0.82370 0.0001 45	1.00000 0.0000 46	-0.18116 0.2392 44	0.63226 0.0001 44	-0.25366 0.0966 44
PKT	0.67845 0.0001 45	0.57799 0.0001 44	0.32929 0.0272 45	0.60689 0.0001 43	0.54105 0.0001 45	0.71440 0.0013 17	0.10052 0.5265 42	0.63974 0.0001 42	0.18146 0.2442 43	-0.18116 0.2392 44	1.00000 0.0000 45	-0.43619 0.0027 45	0.65468 0.0001 45
TIM	-0.12637 0.4081 45	-0.48691 0.0008 44	-0.61288 0.0001 45	-0.61705 0.0001 43	-0.52273 0.0002 45	0.11288 0.6662 17	0.05166 0.7453 42	-0.47459 0.0015 42	0.33200 0.0296 43	0.63226 0.0001 44	-0.43619 0.0027 45	1.00000 0.0000 45	-0.32232 0.0308 45
DT	0.54653 0.0001 45	0.50379 0.0005 44	0.30857 0.0392 45	0.50726 0.0005 43	0.35833 0.0156 45	0.41591 0.0968 17	0.41461 0.0063 42	0.46323 0.0020 42	-0.10450 0.5048 43	-0.25366 0.0966 44	0.65468 0.0001 45	-0.32232 0.0308 45	1.00000 0.0000 45

Table C3. Correlation matrix for LAN paste specimens (1985-86)

PEARSON CORRELATION COEFFICIENTS / PROB > |R| UNDER HO:RHO=0 / NUMBER OF OBSERVATIONS

	H4	D1	D7	D14	D28	D56	ACE	HCE	IS	FS	PKT	TIM	DT
H4	1.00000 0.0000 29	0.82218 0.0001 29	0.80196 0.0001 29	0.81858 0.0001 29	0.84535 0.0001 29	0.88306 0.0007 10	-0.34114 0.0881 26	0.12402 0.5377 27	-0.18327 0.3913 24	-0.68966 0.0002 24	0.73837 0.0001 22	-0.25088 0.2601 22	0.78112 0.0001 22
D1	0.82218 0.0001 29	1.00000 0.0000 30	0.88661 0.0001 29	0.75855 0.0001 29	0.82421 0.0001 30	0.72546 0.0176 10	-0.18382 0.3587 27	0.14491 0.4619 28	-0.16512 0.4303 25	-0.55683 0.0039 25	0.67035 0.0005 23	-0.47509 0.0220 23	0.72483 0.0001 23
D7	0.80196 0.0001 29	0.88661 0.0001 29	1.00000 0.0000 29	0.79580 0.0001 29	0.85576 0.0001 29	0.68696 0.0282 10	-0.18744 0.3592 26	0.23728 0.2334 27	-0.11141 0.6043 24	-0.53770 0.0067 24	0.63491 0.0015 22	-0.44758 0.0367 22	0.65151 0.0010 22
D14	0.81858 0.0001 29	0.75855 0.0001 29	0.79580 0.0001 29	1.00000 0.0000 29	0.88191 0.0001 29	0.78373 0.0073 10	-0.39569 0.0454 26	0.38106 0.0499 27	-0.20541 0.3356 24	-0.55991 0.0045 24	0.76467 0.0001 22	-0.32984 0.1338 22	0.73467 0.0001 22
D28	0.84535 0.0001 29	0.82421 0.0001 30	0.85576 0.0001 29	0.88191 0.0001 29	1.00000 0.0000 31	0.65810 0.0386 10	-0.40609 0.0320 28	0.37050 0.0479 29	0.04585 0.8240 26	-0.48449 0.0121 26	0.64019 0.0008 24	-0.46774 0.0212 24	0.71319 0.0001 24
D56	0.88306 0.0007 10	0.72546 0.0176 10	0.68696 0.0282 10	0.78373 0.0073 10	0.65810 0.0386 10	1.00000 0.0000 10	-0.17987 0.6190 10	0.44959 0.1924 10	0.44201 0.2009 10	0.46478 0.1759 10	0.61244 0.0598 10	-0.39605 0.2572 10	0.72833 0.0169 10
ACE	-0.34114 0.0881 26	-0.18382 0.3587 27	-0.18744 0.3592 26	-0.39569 0.0454 26	-0.40609 0.0320 28	-0.17987 0.6190 10	1.00000 0.0000 28	-0.32171 0.0950 28	0.00748 0.9730 23	-0.01435 0.9482 23	-0.17085 0.4471 22	-0.09599 0.6709 22	-0.02426 0.9146 22
HCE	0.12402 0.5377 27	0.14491 0.4619 28	0.23728 0.2334 27	0.38106 0.0499 27	0.37050 0.0479 29	0.44959 0.1924 10	-0.32171 0.0950 28	1.00000 0.0000 29	0.27308 0.1967 24	0.10972 0.6098 24	0.27571 0.2143 22	-0.29583 0.1813 22	0.23634 0.2896 22
IS	-0.18327 0.3913 24	-0.16512 0.4303 25	-0.11141 0.6043 24	-0.20541 0.3356 24	0.04585 0.8240 26	0.44201 0.2009 10	0.00748 0.9730 23	0.27308 0.1967 24	1.00000 0.0000 26	0.24091 0.2358 26	0.21369 0.3523 21	-0.25460 0.2654 21	0.27194 0.2331 21
FS	-0.68966 0.0002 24	-0.55683 0.0039 25	-0.53770 0.0067 24	-0.55991 0.0045 24	-0.48449 0.0121 26	0.46478 0.1759 10	-0.01435 0.9482 23	0.10972 0.6098 24	0.24091 0.2358 26	1.00000 0.0000 26	-0.72662 0.0002 21	0.01198 0.9589 21	-0.68626 0.0006 21
PKT	0.73837 0.0001 22	0.67035 0.0005 23	0.63491 0.0015 22	0.76467 0.0001 22	0.64019 0.0008 24	0.61244 0.0598 10	-0.17085 0.4471 22	0.27571 0.2143 22	0.21369 0.3523 21	-0.72662 0.0002 21	1.00000 0.0000 24	-0.42838 0.0367 24	0.90813 0.0001 24
TIM	-0.25088 0.2601 22	-0.47509 0.0220 23	-0.44758 0.0367 22	-0.32984 0.1338 22	-0.46774 0.0212 24	-0.39605 0.2572 10	-0.09599 0.6709 22	-0.29583 0.1813 22	-0.25460 0.2654 21	0.01198 0.9589 21	-0.42838 0.0367 24	1.00000 0.0000 24	-0.46949 0.0206 24
DT	0.78112 0.0001 22	0.72483 0.0001 23	0.65151 0.0010 22	0.73467 0.0001 22	0.71319 0.0001 24	0.72833 0.0169 10	-0.02426 0.9146 22	0.23634 0.2896 22	0.27194 0.2331 21	-0.68626 0.0006 21	0.90813 0.0001 24	-0.46949 0.0206 24	1.00000 0.0000 24

Table C4. Correlation matrix for OTT paste specimens (1985-88)

PEARSON CORRELATION COEFFICIENTS / PROB > |R| UNDER HO:RHO=0 / NUMBER OF OBSERVATIONS

	H4	D1	D3	D7	D14	D28	ACE	HCE	IS	FS	PKT	TIM	DT
H4	1.00000	0.43193	0.22793	0.18024	0.20144	0.15580	-0.22076	-0.09611	-0.12512	-0.19741	0.34480	0.27244	0.58892
	0.0000	0.0001	0.0477	0.0929	0.0598	0.1545	0.0521	0.3964	0.2510	0.0685	0.0012	0.0112	0.0001
	88	81	76	88	88	85	78	80	86	86	86	86	86
D1	0.43193	1.00000	0.90259	0.76609	0.77599	0.78123	-0.34708	0.59430	-0.08484	-0.20470	0.15874	-0.07179	0.27305
	0.0001	0.0000	0.0001	0.0001	0.0001	0.0001	0.0010	0.0001	0.4037	0.0421	0.1224	0.4870	0.0071
	81	101	72	100	99	99	87	93	99	99	96	96	96
D3	0.22793	0.90259	1.00000	0.91647	0.83810	0.91845	-0.49996	0.75509	0.11093	-0.20407	0.08237	-0.16207	0.23345
	0.0477	0.0001	0.0000	0.0001	0.0001	0.0001	0.0001	0.0001	0.3401	0.0770	0.4763	0.1591	0.0410
	76	72	78	78	78	77	70	73	76	76	77	77	77
D7	0.18024	0.76609	0.91647	1.00000	0.90593	0.95621	-0.42702	0.60487	-0.06813	-0.26153	0.05976	-0.20649	0.17839
	0.0929	0.0001	0.0001	0.0000	0.0001	0.0001	0.0001	0.0001	0.4878	0.0068	0.5508	0.0373	0.0728
	88	100	78	108	106	105	93	99	106	106	102	102	102
D14	0.20144	0.77599	0.83810	0.90593	1.00000	0.93137	-0.46576	0.67111	-0.07307	-0.23162	0.09985	-0.22891	0.21584
	0.0598	0.0001	0.0001	0.0001	0.0000	0.0001	0.0001	0.0001	0.4588	0.0174	0.3205	0.0213	0.0302
	88	99	78	106	107	104	93	98	105	105	101	101	101
D28	0.15580	0.78123	0.91845	0.95621	0.93137	1.00000	-0.45373	0.66846	-0.08040	-0.26609	0.00672	-0.24100	0.14947
	0.1545	0.0001	0.0001	0.0001	0.0001	0.0000	0.0001	0.0001	0.4172	0.0063	0.9471	0.0157	0.1377
	85	99	77	105	104	106	93	99	104	104	100	100	100
ACE	-0.22076	-0.34708	-0.49996	-0.42702	-0.46576	-0.45373	1.00000	-0.19219	-0.26413	-0.07538	0.02448	-0.12550	-0.11348
	0.0521	0.0010	0.0001	0.0001	0.0001	0.0001	0.0000	0.0664	0.0110	0.4751	0.8199	0.2412	0.2897
	78	87	70	93	93	93	94	92	92	92	89	89	89
HCE	-0.09611	0.59430	0.75509	0.60487	0.67111	0.66846	-0.19219	1.00000	-0.08427	-0.23945	-0.15013	-0.37810	-0.12149
	0.3964	0.0001	0.0001	0.0001	0.0001	0.0001	0.0664	0.0000	0.4094	0.0176	0.1465	0.0002	0.2409
	80	93	73	99	98	99	92	100	98	98	95	95	95
IS	-0.12512	-0.08484	0.11093	-0.06813	-0.07307	-0.08040	-0.26413	-0.08427	1.00000	0.74173	-0.29774	0.50165	-0.34810
	0.2510	0.4037	0.3401	0.4878	0.4588	0.4172	0.0110	0.4094	0.0000	0.0001	0.0025	0.0001	0.0004
	86	99	76	106	105	104	92	98	107	107	101	101	101
FS	-0.19741	-0.20470	-0.20407	-0.26153	-0.23162	-0.26609	-0.07538	-0.23945	0.74173	1.00000	-0.26834	0.69496	-0.35881
	0.0685	0.0421	0.0770	0.0068	0.0174	0.0063	0.4751	0.0176	0.0001	0.0000	0.0067	0.0001	0.0002
	86	99	76	106	105	104	92	98	107	107	101	101	101
PKT	0.34480	0.15874	0.08237	0.05976	0.09985	0.00672	0.02448	-0.15013	-0.29774	-0.26834	1.00000	-0.18226	0.71196
	0.0012	0.1224	0.4763	0.5508	0.3205	0.9471	0.8199	0.1465	0.0025	0.0067	0.0000	0.0654	0.0001
	86	96	77	102	101	100	89	95	101	101	103	103	103
TIM	0.27244	-0.07179	-0.16207	-0.20649	-0.22891	-0.24100	-0.12550	-0.37810	0.50165	0.69496	-0.18226	1.00000	-0.19243
	0.0112	0.4870	0.1591	0.0373	0.0213	0.0157	0.2412	0.0002	0.0001	0.0001	0.0654	0.0000	0.0515
	86	96	77	102	101	100	89	95	101	101	103	103	103
DT	0.58892	0.27305	0.23345	0.17839	0.21584	0.14947	-0.11348	-0.12149	-0.34810	-0.35881	0.71196	-0.19243	1.00000
	0.0001	0.0071	0.0410	0.0728	0.0302	0.1377	0.2897	0.2409	0.0004	0.0002	0.0001	0.0515	0.0000
	86	96	77	102	101	100	89	95	101	101	103	103	103

APPENDIX D: OGS and LGS TEST RESULTS

Description of Variables in Table D1

Many of the variables listed in Table D1 have been already been described in Appendix B and Appendix C. Hence, the reader must refer to the previous listings for a detailed description of of these variables. However, Table D1 also contains seven variables that have not been defined earlier. These variables have to do with mineralogical information obtained using x-ray diffraction. The variables can be defined as follows.

Q	=	intensity of the 22% diffraction peak for Quartz (net counts per second, ncps)
UNK	=	intensity of a unassigned diffraction peak (ncps)
CAS	=	intensity of the 100% diffraction peak for tetracalcium-trialuminate-sulfate (ncps)
AN	=	intensity of the 100% diffraction peak for Anhydrite (ncps)
C3A-1	=	intensity of the 100% diffraction peak for tricalcium aluminate (ncps)
L	=	intensity of the 100% diffraction peak for Lime (ncps)
P	=	intensity of the 100% diffraction peak for Periclase (ncps)
.	=	missing data or peak too small to measure accurately

Table D1. Raw data for LGS and OGS paste specimens

Sample Name	Day No. (7/1/87)	Day No. (1/1/83)	4 hour	1 day	3 day	7 day	14 day	28 day	56 day	%exp air	%exp humid	Initial set	Final set	Onset time	Final time	ΔTemp	Peak Temp
LGS070987	0009	1651	318	988	•	2517	2538	4289	3110	•	•	9.000	13.0	15.0	39.0	2.5	•
LGS071587	0015	1657	931	1417	•	3259	4108	3410	4196	-0.12	0.087	7.000	9.0	21.0	35.0	6.5	31.5
LGS072187	0021	1663	476	1139	•	3737	3853	3202	3812	-0.122	0.056	10.000	12.0	23.0	35.0	4.8	29.8
LGS072987	0029	1671	702	1796	•	2955	4094	3849	3900	•	0.098	7.000	10.0	13.0	37.0	4.0	28.5
LGS080487	0035	1677	624	1554	•	2105	4322	3897	4094	•	0.094	8.000	12.0	15.0	36.0	3.0	26.0
LGS081287	0043	1685	232	368	•	1267	2348	2241	2364	-0.055	0.070	7.000	10.0	9.0	20.0	2.8	27.8
LGS082087	0051	1693	327	730	•	1747	2578	3459	2978	•	0.150	4.500	5.5	7.0	19.0	4.5	28.5
LGS082687	0057	1699	648	1392	•	3088	3928	4458	4088	•	0.073	7.000	10.0	13.0	34.0	6.3	31.3
LGS090487	0066	1708	531	1430	•	3523	3596	2999	4121	•	0.085	7.000	10.0	14.0	30.0	4.3	28.3
LGS091087	0072	1714	524	755	•	2720	3653	3369	2862	•	0.010	10.000	14.0	12.0	26.0	3.3	26.8
LGS091687	0078	1720	661	822	•	3515	3844	3545	4408	-0.156	0.028	12.000	17.0	13.0	35.0	2.8	29.8
LGS092087	0082	1724	671	1001	•	3540	4499	3510	4066	-0.186	0.018	8.000	11.0	10.0	23.0	6.3	30.3
LGS092487	0086	1728	936	1746	•	3893	4337	3362	4368	•	0.037	8.000	11.0	10.0	27.0	7.8	31.3
LGS092687	0088	1730	829	1625	•	3458	3559	4701	4832	•	0.000	12.000	19.0	14.0	47.0	5.2	29.2
LGS103087	0122	1764	280	1103	•	2242	2834	3239	3752	-0.061	0.128	6.000	8.0	9.0	18.0	3.8	27.3
LGS110187	0124	1766	300	646	•	1985	1593	2944	3288	•	0.088	7.000	11.0	14.0	19.0	3.5	26.5
LGS110387	0126	1768	259	862	•	2767	1940	3526	3853	•	0.111	7.000	10.0	15.0	20.0	3.4	26.4
LGS110587	0128	1770	230	832	•	2738	1817	3253	3471	•	0.107	9.000	14.0	25.0	21.0	2.3	25.3
OGS070887	0008	1650	280	423	•	914	970	1395	1483	-0.061	-0.017	23	27	•	25	4.5	29.5
OGS071587	0015	1657	399	793	•	1087	1571	1408	2076	•	-0.011	24	29	•	34	4.3	28.3
OGS072287	0022	1664	566	628	•	895	919	1112	1256	-0.046	0.009	10	14	20	26	6.8	29.8
OGS072987	0029	1671	305	490	•	818	876	1014	1211	-0.036	-0.020	11.0	15.0	•	23.0	4.3	28.3
OGS080587	0036	1678	364	512	•	778	893	806	972	-0.038	0.002	19.0	38.0	•	24.0	5.0	29.0
OGS081287	0043	1685	249	363	•	520	590	696	796	•	•	43.0	57.0	40.0	48.0	3.8	26.8
OGS081987	0050	1692	290	394	•	483	497	704	743	-0.042	•	22.0	36.0	•	30.0	4.0	28.0
OGS082687	0057	1699	550	723	•	886	1004	984	1161	-0.076	-0.011	12.000	30.0	•	46.0	3.8	26.8
OGS090287	0064	1706	498	558	•	730	770	777	1076	-0.074	0.002	9.000	21.0	26.0	38.0	6.5	27.5
OGS090987	0071	1713	434	577	•	666	691	809	1041	•	•	11.000	16.0	•	28.0	6.5	29.5
OGS091487	0076	1718	390	496	•	600	788	902	956	-0.064	0.009	10.000	16.0	23.0	34.0	6.2	27.2
OGS091687	0078	1720	115	247	•	384	406	412	435	•	•	19.000	27.0	34.0	47.0	3.5	26.5
OGS091887	0080	1722	79	158	•	238	258	236	229	•	•	33.000	51.0	31.0	52.0	2.5	24.2
OGS092187	0083	1725	316	501	•	735	863	675	1095	-0.057	0.010	15.000	21.0	36.0	56.0	1.8	24.8
OGS092387	0085	1727	306	356	•	552	597	692	845	•	-0.001	14.000	23.0	18.0	27.0	7.3	30.2
OGS092587	0087	1729	328	408	•	670	762	738	807	-0.037	-0.007	10.000	14.0	21.0	26.0	4.5	27.5
OGS100687	0098	1740	33	173	•	347	265	240	532	-0.026	-0.005	94.000	178.0	9.0	15.0	0.5	22.5
OGS100787	0099	1741	•	•	•	•	•	•	•	•	•	97.000	198.0	6.0	12.0	0.3	22.8
OGS100887	0100	1742	237	360	•	613	1577	2277	2591	-0.034	•	23.000	27.0	19.0	42.0	3.0	26.0
OGS100987	0101	1743	199	298	•	1793	1996	2169	3342	-0.029	•	35.000	41.0	9.0	20.0	0.5	23.5
OGS101287	0104	1746	207	621	•	2273	2508	2030	2991	-0.035	•	11.000	15.0	7.0	14.0	1.3	24.8

Table D1. (Continued)

Sample Name	SrO	MgO	Na2O	Fe2O3	SO3	BaO	MnO	SiO2	CaO	K2O	P2O5	Al2O3	Peak Intensity Q	UNK	CAS	AN	C3A-1	L	P
LGS070987	0.458	4.381	1.136	5.848	1.552	0.628	0.026	37.753	24.136	0.419	1.434	17.822	117	23	25	48	132	51	46
LGS071587	0.432	4.577	1.346	6.093	2.333	0.607	0.030	33.334	26.628	0.310	1.277	18.593	96	19	30	67	170	104	78
LGS072187	0.440	4.435	1.706	5.952	1.898	0.660	0.035	32.525	25.618	0.388	1.337	19.443	49	14	24	52	174	55	52
LGS072987	0.440	4.228	1.728	5.841	1.880	0.637	0.030	33.451	25.503	0.392	1.257	19.000	58	20	27	64	172	59	51
LGS080487	0.441	4.561	1.359	6.124	1.881	0.656	0.030	33.009	26.016	0.341	1.384	19.063	56	20	29	60	160	64	60
LGS081287	0.439	3.981	1.404	6.169	1.473	0.656	0.029	34.529	23.258	0.434	1.442	19.355	60	30	17	58	140	25	44
LGS082087	0.418	4.299	1.669	5.828	1.859	0.616	0.030	33.968	25.214	0.364	1.148	18.728	65	13	33	71	188	61	79
LGS082687	0.410	5.043	1.569	5.662	2.085	0.635	0.028	35.465	25.870	0.296	0.950	17.047	62	17	29	78	194	88	99
LGS090487	0.396	5.237	1.619	6.144	2.093	0.629	0.030	36.503	25.542	0.284	0.775	16.004	80	17	23	77	136	73	95
LGS091087	0.389	5.305	1.594	6.386	2.236	0.677	0.036	36.085	25.891	0.286	0.860	15.549	85	18	23	77	168	64	104
LGS091687	0.376	5.319	1.508	7.025	2.610	0.662	0.035	35.298	26.584	0.230	0.768	14.852	62	17	25	87	140	101	113
LGS092087	0.409	5.870	1.631	6.641	2.639	0.680	0.032	35.142	26.748	0.240	0.892	15.022	53	12	25	85	161	79	125
LGS092487	0.399	5.368	1.691	6.662	2.701	0.642	0.035	33.410	27.127	0.215	0.778	15.332	55	18	23	91	172	99	125
LGS092687	0.405	5.380	1.590	6.954	3.180	0.645	0.034	33.023	27.779	0.239	0.829	15.827	44	21	31	111	159	97	126
LGS103087	0.494	4.975	1.375	6.312	2.041	0.731	0.028	34.254	26.148	0.316	1.571	17.005	48	20	22	72	142	73	74
LGS110187	0.470	4.303	1.351	6.030	1.560	0.708	0.028	35.752	24.198	0.404	1.420	18.658	63	20	17	48	146	44	72
LGS110387	0.455	4.890	1.398	6.877	1.874	0.678	0.029	35.012	25.495	0.319	1.302	16.851	60	17	22	60	171	45	86
LGS110587	0.446	4.514	1.373	6.249	1.617	0.664	0.027	34.976	24.766	0.364	1.247	18.484	54	17	25	50	150	52	59
OGS070887	0.477	4.461	1.958	5.637	2.321	0.727	0.028	32.202	25.953	0.357	1.666	18.459	39	•	24	79	129	62	54
OGS071587	0.461	4.750	1.936	5.533	2.294	0.665	0.029	32.193	26.458	0.342	1.485	18.466	•	•	32	87	146	59	65
OGS072287	0.424	4.586	2.797	6.249	3.394	0.638	0.032	29.34	27.032	0.295	1.379	18.154	•	•	•	•	•	•	•
OGS072987	0.522	4.349	2.108	5.682	2.309	0.766	0.027	31.912	25.884	0.322	1.955	17.863	37	•	•	67	123	43	64
OGS080587	0.539	4.486	2.609	5.704	2.864	0.814	0.026	30.223	25.723	0.341	2.299	18.293	23	•	•	87	138	58	62
OGS081287	0.497	4.274	2.331	5.936	2.589	0.752	0.028	31.297	24.561	0.381	2.077	18.644	•	•	•	82	118	49	43
OGS081987	0.466	4.379	2.917	5.675	2.673	0.685	0.030	30.884	24.993	0.372	1.665	18.564	•	20	•	77	133	48	55
OGS082687	0.418	4.596	3.228	5.532	3.075	0.617	0.029	31.549	26.199	0.291	1.085	17.662	•	•	•	•	•	•	•
OGS090287	0.412	4.651	3.181	5.485	3.235	0.637	0.026	30.510	26.142	0.293	0.991	18.172	29	•	•	98	158	48	59
OGS090987	0.400	4.657	3.369	5.530	3.504	0.625	0.028	30.245	26.025	0.279	0.839	18.142	•	•	•	•	•	•	•
OGS091487	0.398	4.448	3.321	5.615	3.486	0.637	0.026	29.502	26.667	0.236	0.791	17.830	•	•	•	88	146	72	71
OGS091687	0.405	4.522	3.390	5.581	4.530	0.662	0.029	28.923	26.330	0.247	0.931	17.706	31	•	•	176	150	77	56
OGS091887	0.414	4.424	3.348	5.606	3.627	0.648	0.026	30.520	26.086	0.277	0.970	17.738	50	22	•	170	140	49	44
OGS092187	0.436	4.274	3.062	5.536	4.136	0.661	0.026	31.038	24.834	0.310	1.260	17.280	30	•	26	153	143	45	46
OGS092387	0.414	4.544	2.790	5.540	3.008	0.645	0.027	31.575	25.684	0.315	1.039	18.011	39	•	29	81	138	53	61
OGS092587	0.477	4.457	1.958	5.543	2.321	0.724	0.028	33.303	25.164	0.381	1.692	17.881	61	•	•	71	94	46	54
OGS100687	0.573	4.047	2.089	5.780	1.546	0.824	0.025	33.288	22.611	0.448	2.251	19.777	•	23	•	56	69	31	37
OGS100787	0.561	4.254	2.222	5.675	1.501	0.843	0.026	33.001	23.009	0.463	2.231	20.019	•	•	•	•	•	•	•
OGS100887	0.494	4.462	2.278	5.718	1.981	0.768	0.026	31.588	25.355	0.371	1.695	19.041	•	•	•	52	109	35	39
OGS100987	0.530	4.252	2.213	5.797	1.841	0.791	0.026	32.486	24.244	0.407	1.946	19.389	28	•	•	55	108	38	50
OGS101287	0.443	4.207	2.114	5.554	1.764	0.677	0.026	33.903	23.418	0.455	1.287	19.866	33	•	•	68	96	32	32